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Whey protein concentrate and skimmed milk powder as encapsulation agents for coffee silverskin extracts processed by spray drying

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Research Article

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Abstract

We tested the hypothesis that milk proteins, through microencapsulation, guarantee protection against bioactive substances in coffee silverskin extracts. Therefore, the aim of this study was to carry out technological, nutritional and physicochemical characterisation of a coffee silverskin extract microencapsulated using instant skim milk powder and whey protein concentrate as wall materials. The aqueous extract of coffee silverskin was spray-dried using 10% (w/v) skim milk powder and whey protein concentrate. The samples were characterised by determining the water content, water activity, particle size distribution, colour analysis and total phenolic compound content as well as antioxidant activity using 2,2-diphenyl-radical 1-picrylhydrazyl scavenging methods, nitric oxide radical inhibition and morphological analysis. The product showed water activity within a range that ensured greater stability, and the reduced degradation of the dried coffee silverskin extract with whey protein concentrate resulted in better rehydration ability. The luminosity parameter was higher and the browning index was lower for the encapsulated samples than for the pure coffee silverskin extract. The phenolic compound content (29.23 ± 8.39 and 34.00 ± 8.38 mg gallic acid equivalents/g for the coffee silverskin extract using skimmed milk powder and whey protein concentrate, respectively) and the antioxidant activity of the new product confirmed its potential as a natural source of antioxidant phenolic compounds. We conclude that the dairy matrices associated with spray drying preserved the bioactive and antioxidant activities of coffee silverskin extracts.

Milk is an interesting matrix candidate for encapsulation by spray drying owing to its structural and physicochemical properties, which enable it to control the bioaccessibility of bioactive compounds and promote their bioavailability (Livney, 2010). Skimmed milk powder (SMP) is one of the most common types of protein used in microencapsulation because it is highly efficient, affordable and economical. It has excellent amphiphilic characteristics and offers favourable characteristics for the microencapsulation of bioactive compounds. It has the ability to quickly absorb compounds that form a thickened layer. The hydrophobic composition and flocculation may be composed of electrostatic bonds (Coimbra *et al*., 2020). Whey protein concentrate (WPC) is an excellent wall material for the encapsulation of bioactive ingredients. It presents an excellent ability for film formation, has great retention capability of nutrients in the encapsulation process, and has high nutritional value (Shishir and Chen, 2017).

Coffee production for commercialisation as a raw bean generates approximately 45–50% waste, including coffee silverskin (Gemechu, 2020). The composition of this residue has characteristics that favour its application in foods as a natural and sustainable ingredient (Costa *et al*., 2018).

Spray drying is one of the most widely used microencapsulation methods in the food industry. For microcapsules intended for food purposes, the encapsulating wall materials must be food-grade, easy to handle and have low hygroscopicity and biodegradability (Rutz *et al*., 2013). The encapsulation of phenolic compounds and antioxidants is an important strategy for preserving their properties for a longer time, as the encapsulating materials act as barriers to oxygen and water, improving their stability and enabling their use in the food industry (Lavelli *et al*., 2016). This study tested the hypothesis that milk proteins, through microencapsulation, guarantee protection against bioactive substances in coffee silverskin extracts.

The aim was to carry out technological, nutritional, and physicochemical characterisations of coffee silverskin extract microencapsulated using SMP and WPC as wall materials.

Materials and methods

To obtain the coffee silverskin extract, ground and sieved samples were added to water and subjected to heating and constant agitation in a mechanical stirrer (model 715 W; Fisatom, Sao Paulo, Brazil). Subsequently, the extract was filtered and stored at −25°C before analysis. Microencapsulation was performed using a spray dryer (Mini Spray Dryer B-290; Buchi, Flawil, Switzerland) with solutions of encapsulating agents (SMP and WPC) prepared at 10% (w/v).

Water activity (a_w) was measured directly, using an AquaLab 4TE instrument (Decagon Devices, Pullman, WA, USA) at 25 ± 1°C. The particle size distribution of the powders during the rehydration process was obtained using a Beckman Coulter LS 13320 laser diffraction analyser with an aqueous liquid module (Beckman Coulter, Brea, CA, USA). The colours of the powders were determined using a colorimeter (CR-400 Chroma Meter; Konica Minolta, Tokyo, Japan).

Total phenol content was determined using the Folin-Ciocalteu spectrophotometric method. The 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical scavenging method described by Govindarajan *et al.* (2003) was used to determine the antioxidant potential of the samples, with some modifications. Nitric oxide radical scavenging activity was measured indirectly using the Griess method.

The statistical analysis of the results was performed using GraphPad Prism® (version 8.0; GraphPad, San Diego, CA, USA). More detailed information is provided in the Supplementary materials.

Results and Discussion

The coffee silverskin extract and the products obtained are listed in Table 1. The water content of the new product was lower than that of the coffee silverskin extract. Water content is an important characteristic of powdered products because it affects fluidity, stickiness, and stability during storage. In addition, the low water content prevents particle agglomeration and clogging, which can reduce the retention of bioactive components. The water content values obtained for the new products were within the ideal range (less than 5%), which ensured greater stability and reduced the degradation of the powdered products (Carmo *et al.*, 2018).

When comparing the encapsulating materials, the coffee silverskin extract encapsulated with WPC showed a lower a_w than the one encapsulated with SMP ($P \leq 0.05$), and was significantly lower when compared to the freeze-dried coffee silverskin extract. These values were lower than those reported in the study by Calva-Estrada *et al.* (2018), in which microcapsules of natural and synthetic vanilla extract encapsulated with WPC showed a_w values of 0.350 ± 0.01 and 0.335 ± 0.02, respectively. The a_w values we found were also slightly lower than those found by Rocha *et al.* (2019), who reported values between 0.3 and 0.4 for jabuticaba, jussara, and blueberry powders encapsulated with different agents, including WPC, maltodextrin, and gum arabic.

The coffee silverskin extract encapsulated with WPC had a significantly ($P \leq 0.05$) lower Dv90 value than the coffee silverskin extract encapsulated with SMP. A higher Dv90 value is known to indicate a lower powder reconstitution efficiency.

There was no significant difference in the <1 µm (%) parameter ($P \leq 0.05$) between the three samples. A greater number of particles <1 µm, results in a better rehydration capacity of the powder (Francisquini *et al.*, 2020).

There was a significant difference ($P \leq 0.05$) in L^* values between the coffee silverskin extract and the ingredients obtained. These results indicate that encapsulation makes the powder clearer, which is a characteristic that benefits its use as an ingredient in the food industry. For the a^* coordinate, the value found for the coffee silverskin extract encapsulated with WPC was closer to green, when compared to the coffee silverskin extract encapsulated with SMP. When comparing the b^* variable, the results indicated greater proximity of encapsulated coffee silverskin extract samples to the yellow colour when compared to the nonencapsulated coffee silverskin extract.

The browning index is used to evaluate the brown colour intensity of food products. In this study, the browning index value showed no statistically significant difference between the bioactive samples, demonstrating that they are not different in terms of brown colour intensity. However, the coffee silverskin extract significantly differed ($P < 0.05$) from the other samples.

There were no significant differences in phenolic compounds between the encapsulated coffee silverskin extracts. Nzekoue *et al.* (2020) performed coffee silverskin extraction using different solvents, which directly influenced the total phenolic content. In the DPPH assay, an increase in the antioxidant capacity of coffee silverskin extract encapsulated in WPC was observed. The IC_{50} value obtained for the coffee silverskin extract encapsulated with WPC showed a higher antioxidant potential than the values obtained for the freeze-dried coffee silverskin extract and the coffee silverskin extract encapsulated with SMP, and it was statistically equivalent to the value obtained for quercetin, the reference substance in the reaction. This may be attributed to the inactivation of peroxidases, which have prooxidant activity; the formation of new antioxidant compounds, or an improvement in the antioxidant capacity of natural compounds (Gomes *et al.*, 2021). For the nitric oxide radical scavenging activity, there was a significant difference ($P \leq 0.05$) between the encapsulated coffee silverskin extracts.

In general, antioxidant activity can be increased by the conjugation of proteins with phenolic compounds. However, some studies have reported that this reduces antioxidant activity. Thus, the increase in antioxidant activity when associating milk proteins with the phenolic compounds present in the coffee silverskin extract using the DPPH method can be explained by the synergistic interaction between the molecules.

The coffee silverskin extract encapsulated in WPC had a better rehydration capacity than the extract encapsulated in SMP. However, the morphologies of both products were very similar (online Supplementary Fig. S1). There was no significant difference in the phenolic compounds among the encapsulated coffee silverskin extracts; however, an increase in antioxidant capacity was observed for the coffee silverskin extract encapsulated with WPC.

In relation to the parameters analysed, it can be concluded that the extract encapsulated with WPC showed better results than the extract encapsulated with SMP, as it showed greater a_w, rehydration capacity and antioxidant activity, which make it more favourable for development as an ingredient.

In conclusion, the coffee silverskin extract encapsulated with SMP and WPC by spray drying was more stable than the pure freeze-dried extract, thus promoting better conservation and extending the shelf life. From the analysis of the particle size

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Table 1. Analysis of physicochemical properties, particle size distribution, colorimetric parameters of powders, phenolic compounds and antioxidant capacity

	E	*E*/SMP	*E*/WPC	Quercetin*/gallic acid**
Moisture (%)	9.83 ± 2.33[a]	4.02 ± 0.26[b]	4.77 ± 0.39[b]	–
Water activity (a_w)	0.44 ± 0.00[a]	0.24 ± 0.01[b]	0.20 ± 0.01[c]	–
Dv90 (μm)	31.57 ± 0.37[a]	196.17 ± 7.71[b]	18.61 ± 7.46[a]	–
<1 μm (%)	9.50 ± 0.35[a]	6.92 ± 1.84[a]	9.07 ± 4.12[a]	–
*L**	31.16 ± 1.16[a]	88.47 ± 0.22[b]	88.21 ± 0.05[b]	–
*a**	9.93 ± 0.20[a]	0.75 ± 0.37[b]	−0.07 ± 0.07[c]	–
*b**	13.02 ± 2.56[a]	15.90 ± 0.30[b]	15.35 ± 0.14[b]	–
BI	77.36 ± 16.65[a]	19.99 ± 0.40[b]	18.60 ± 0.14[b]	–
Phenolic compounds (mg GAE/g)	74.78 ± 5.02[a]	29.23 ± 8.39[b]	34.00 ± 8.38[b]	–
DPPH (IC50)	66.15 ± 6.66[a]	184.35 ± 33.85[b]	9.53 ± 2.92[c]	0.053 ± 0.00[c]
NO (% inhibition at the concentration 250 μg/ml)	59.40 ± 3.15[a]	57.73 ± 3.05[ab]	44.24 ± 0.76[c]	51.74 ± 3.09[b]

Values expressed as mean ± standard deviation. Means followed by the same letter does not differ statistically in the Turkey test at the level of 5% stability ($P \leq 0.05$), applied to the same line. *E*, freeze-dried coffee silverskin extract; *E*/SMP, coffee silverskin extract encapsulated with skimmed milk powder; *E*/WPC, coffee silverskin extract encapsulated with whey protein concentrate. *L** indicates lightness, *a** coordinate green-red, *b** coordinate blue-yellow and BI browning index. *Standard used as a reference in DPPH assay; **Standard used as a reference in NO assay.

distribution, it can be inferred that the coffee silverskin extract encapsulated with WPC provides better rehydration capacity, favouring the technological characteristics of the ingredient. In this study, we developed a dried product containing bioactive compounds extracted from coffee silverskin associated with protein matrices by spray drying with promising properties to increase the stability and bioavailability of phenolic substances and produce desirable technological characteristics.

Supplementary material. The supplementary material for this article can be found at https://doi.org/10.1017/S0022029924000128

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