SEMEAR: REVISTA DE ALIMENTAÇÃO, NUTRIÇÃO E SAÚDE

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Multi cyclic extraction of polyphenols from vegetable residue-based flour by means of hydroalcoholic and ultrasound

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Resumo

Há um interesse crescente pela extração de compostos bioativos de resíduos vegetais por métodos mais ecológicos como a extração assistida por ultrassom. O objetivo deste estudo foi extrair polifenóis aplicando uma extração hidroalcoólica com ciclos consecutivos (HAE) ou extração assistida por ultrassom (UAE) na Farinha de Frutas e Hortaliças (FVF), seguida de secagem por aspersão. A UAE modificou bastante a microestrutura do FVF, aumentando a liberação de polifenóis em aproximadamente 215 a 300% no primeiro ciclo de extração. O teor total de polifenóis (TPC) variou de 76,28 ± 2,17 a 92,32 ± 5,79 mg GAE.g^{-1} FVF para HAE e 113,02 ± 2,71 a 134,48 ± 1,66 mg GAE.g^{-1} FVF. As cápsulas tiveram TPC variado de 0,54 ± 0,04 a 1,92 ± 0,04 mg GAE.g^{-1} de pó, com morfologia esférica. O processo em ciclos por ultrassom extraiu aproximadamente 330% mais polifenóis do FVF do que dados relatados anteriormente demonstrando que o FVF tem maior potencial antioxidante a ser explorado.
Palavras-chave: extração sólido-líquido; extração física; resíduos vegetais.

Abstract

There is an increasing interest for the extraction of bioactive compounds from vegetable residues by greener methods as ultrasound-assisted extraction. The aim of this study was to extract polyphenols applying a multi cyclic hydro alcoholic extraction (HAE) or ultrasound-assisted extraction (UAE) on the Fruit and Vegetables Flour (FVF), followed by spray drying. The UAE greatly modified the microstructure of the FVF, increasing the release of polyphenols in approximately 215-300% on first cycle of extraction. The total polyphenol content (TPC) varied from 76.28 ± 2.17 to 92.32 ± 5.79 mg GAE.g^{-1} FVF for HAE and 113.02 ± 2.71 to 134.48 ± 1.66 mg GAE.g^{-1} FVF. The capsules had TPC varied from 0.54 ± 0.04 to 1.92 ± 0.04 mg GAE.g^{-1} of powder, with spherical morphology. The multi cyclic UAE extracted approximately 330% more polyphenols from FVF previous reported data demonstrating that FVF have higher antioxidant potential to be explored.
Keywords: *solid-liquid extraction; physical extraction; vegetable residues*

INTRODUCTION

On the last decades, a growing interest on extracting bioactive compounds from vegetable residues has surged since vegetable waste are a global problem that cost billions of dollars per year to manage while they are also a valuable source of bioactive compounds (1).

Most of bioactive compounds are produced by secondary metabolism of vegetable cells and may be present as extractable polyphenols (or free polyphenols) and non-extractable polyphenols (or bound polyphenols) (2), with the second group forming complexes with starch and non-starch polysaccharides such as cellulose, hemicellulose, pectin, and proteins (3). The polyphenols are bound mostly by hydrogen bounds given by their hydroxyl groups and hydrophobic interactions on the surface of the polymers or in specific sites on the microstructure of these polymers (3).

In order to extract polyphenols, different solid-liquid extractions can be performed, like the conventional method of solvent extraction (2,3) and other novel technologies such as ultrasound (4).

The use of solvents polyphenol extraction aims to reduce the affinity of the polyphenols with the polymer allowing the extraction (3). In addition, is relevant to observe the polymeric composition of the vegetable matrix when extracting bound polyphenols since they can interact with proteins and non-protein polymeric structure. On this way, enzymatic treatments can increase the extraction of bound polyphenols when used as pre-treatments on the matrix, modifying their microstructure and polymeric composition, increasing the extraction is less time (2,5).

The vegetable cell structures are also affected by ultrasound for solid-liquid extraction of polyphenols due to the acoustic cavitation effect, creating the mechanical stress that breaks the polymers and create micro pores, releasing the polyphenols on the medium (6).

Considering the complexity of vegetable polymers and that the type of biding with polyphenols impact the extraction capacity, one single extraction of polyphenols can result in a waste of bound polyphenols varying from 24% to 85% of the total polyphenol content (2). This suggest that multiple extraction cycles can increase the polyphenol extraction and, to improve the extraction of polyphenols from the vegetable matrix, Yang et al (7) applied three consecutive cycles of conventional extraction with 80% aqueous acetone. To reach the same,

de Souza et al (8) applied three cycles of UAE on passion fruit rind.

Since polyphenols are sensible to oxidation, an encapsulation process such as spray drying is recommended. For this purpose, maltodextrin is frequently used as wall material due to its resistance to great water activity variations and the ability to prevent oxidation by reducing the oxygen permeability (9).

Following the tendency of waste valorisation, Ferreira et al. (10) characterized a Fruit and Vegetables Flour (FVF) made from solid waste of an isotonic drink production (10,11). The isotonic drink uses Selecta orange (*Citrus sinensis*), passion fruit (*Passiflora edulis*), watermelon (*Citrullus lanatus*), lettuce (*Lactuca sativa*), courgette (*Cucúrbita pepo*), carrot (*Daucus carota*), spinach (*Spinacea oleracea*), mint (*Mentha s.p.*), taro (*Colocasia esculenta*), cucumber (*Cucumis sativus*) and rocket (*Eruca sativa*) as ingredients. The FVF final composition have 26% of carbohydrates, 9.5% of proteins, 5% of lipids, 11,1% of moisture and ash (10). Also, 48.4% of the biopolymers present on the FVF are dietary fibre, characterized as cellulose, hemicellulose, soluble lignin, insoluble lignin and resistant starch, where 80% are insoluble fibre (12). The FVF presented 22.49 ± 1.59 mg GAE.g^{-1} of total polyphenol content (TPC), and the hydro alcoholic extraction with 75% ethanol solution was the best extraction type in terms of total polyphenol content for a single extraction of polyphenols on this matrix (13). In addition, 88 compounds were also identified (28 were phenolic acids, 32 flavonoids and 28 other polyphenols), from which hesperidin was the most abundant compound (14).

The aim of this study was to evaluate the extraction of polyphenols compounds applying a continuous hydro alcoholic extraction or ultrasound-assisted extraction on the Fruit and Vegetables Flour, using enzymatic process, followed by encapsulation by spray drying.

MATERIAL AND METHODS

The Fruit and Vegetables Flour (FVF) made of Selecta orange (*Citrus sinensis*), passion fruit (*Passiflora edulis*), watermelon (*Citrullus lanatus*), lettuce (*Lactuca sativa*), courgette (*Cucúrbita pepo*), carrot (*Daucus carota*), spinach (*Spinacea oleracea*), mint (*Mentha s.p.*), taro (*Colocasia esculenta*), cucumber (*Cucumis sativus*) and rocket (*Eruca sativa*) mixed solid residue obtained as described by Ferreira et al. (10) was used as matrix on this study. The Viscozyme® and the chemicals used were acquired in Sigma Aldrich.

Extraction process

Hydro alcoholic extraction (HAE)

This extraction was performed with ethanol 75% as extractor at 40°C for 24 h. The supernatant was recovered and the residue was submitted to a new extraction with equal volume of extractor. Three different conditions were applied as follows, with solid:liquid ratio of 1:15 (m:v):

A. FVF: ETOH = (1 g:15 mL);
B. FVF: Viscozyme®:ETOH = (1 g):25 µL*:15 mL)
C. FVF: PC**:Viscozyme®:ETOH = (1 g:1 g:25 µL*: 30 mL)

* - Added on each cycle. ** - *In natura* pineapple crown (PC) was cut in pieces with 0.5cm^2. Each condition was made on triplicate.

Ultrasound-assisted extraction (UAE)

Sample was placed in solution with distilled water on a Dubnoff NT232 water bath (Novatécnica, São Paulo, Brazil) at 30°C for 30 min. After that, the solution was exposed continuously to ultrasonic waves (4) on bench top ultrasound UIP1000hdT of 20 kHz (Hielscher, Teltow, Germany) adjusted to 500 W and 100% amplitude (15). The process occurred for 8 min limiting temperature at 60°C using an ice bath. The solution was filtered in filter paper C41 110 mm (ForLab Express, Rio de Janeiro, Brazil) and the recovered solid residue was submitted to a new extraction with equal volume of extractor. The process was considered finished when the total phenolic compounds did not presented statistical difference ($P < 0.05$) on three consecutive extractions. Three different conditions were applied as mentioned above.

Encapsulation of FVF extracts by spray-dried

Feed solution preparation (FD)

The solution to be encapsulated (SC) was obtained from the mix of all volume of each extraction cycle. The final cycle was considered the first of three cycles that did not presented statistical difference ($P < 0.05$) in TPC amount. The final volume of SC of each process was 270mL for HAE.I; 315mL for HAE.II; 540mL for HAE.III; 900mL for UAE.I; 750mL for

UAE.II; and 1500mL for UAE.III. For the encapsulation, 80 mL of SC and 160 mL of distilled water where homogenized with maltodextrin 10 DE (Corn Products, Mogi-Guaçu, Brazil) that was added until the solution reached 28°Brix (FD).

Encapsulation process

The solutions were dried on a mini spray dryer Büchi B-190 (Büchi, Flawil, Switzerland) following these conditions: 60 mBar vacuum, 75 lb/pol^2 of pressure, 170 °C of inlet temperature and 90°C of outlet temperature (16). The powders were stored in laminated bags at ambient condition. The powder yield was determined as described by De Sá Mendes et al. (2019). Physical properties (moisture, microstructure and density) were analysed in triplicate, while the total polyphenol content was in quintuplicate.

Moisture

The moisture was analysed on an infrared moisture analyser IV2000 (Gehaka, São Paulo, Brazil) (18).

Density analysis

The bulk density (ρ_b) and tap density (ρ_t) as well as the flowability [Carr index (CI)] and cohesiveness [Hausner Correlation (HC)] were determined according to De Sá Mendes et al. (2019). The powder flowability was considered as follows: CI < 15%: great flowability; CI between 15% and 20%: good flowability; CI between 20% and 35%: intermediary flowability; CI between 35% and 45%: low flowability; CI > 45%: very low flowability. On the other hand, their cohesiveness was considered as follows: HC < 1.2: low cohesiveness; HC between 1.2 and 1.4: intermediary cohesiveness; HC > 1.5: high cohesiveness.

Total polyphenol content (TPC)

The total polyphenol content followed the Folin-Ciocalteu technique (17) for each supernatant recovered from the cycles of each extraction process. The analysis was conducted on the Victor Nivo Microplate Reader (Perkin Elmer, German). The results expressed as milligram of Gallic Acid Equivalent per gram of sample (mg GAE g^{-1}). This analysis was made in five replicates.

Scanning Electron Microscopy (SEM)

The FVF residues of each cycle and each condition were oven dried (60 ºC) and observed on a Tabletop Scanning Electron Microscope TM3000 (Hitachi, Tokyo, Japan) and observed on six random points at 15 kV (19).

Statistical analysis

The results were expressed by mean ± standard deviation. Statistical differences between samples were calculated using Student's *t* test for independent samples (Microsoft Excel, Microsoft Corporation, Washington, USA). Results were considered significant at 95% confidence level ($P < 0.05$).

RESULTS AND DISCUSSION

Extraction process

The process was considered finished when the total phenolic compounds of the extract did not present statistical difference ($P < 0.05$) on three consecutive extractions (Figure 1). Eight cycles were necessary for obtaining HAE.I and HAE.III and nine for HAE.II (Figure 1.A). The HAE presented increase of polyphenols extract up to fifth cycle. After that, the Viscozyme® effect on the polymeric structure could be seen on HAE.II, creating significant difference from conditions I and III.

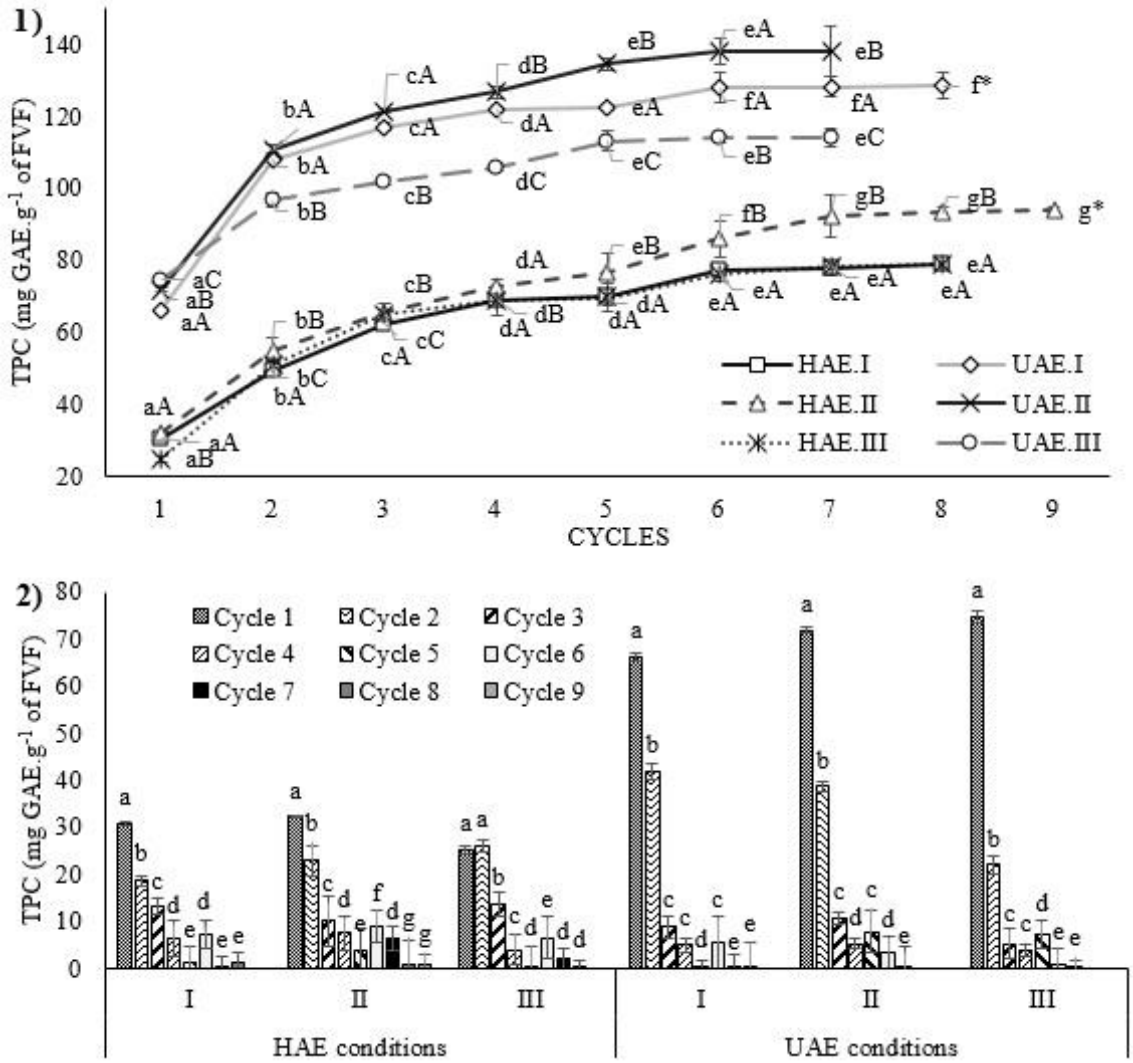

Figure 1: Total polyphenol content (mg GAE.g^{-1} FVF) on different extraction cycles of FVF by hydro alcoholic extraction [HAE - 75% (v/v)] and ultrasound-assisted extraction [UAE], on three different conditions (I, II, III) at solid:liquid ratio on 1:15 (m:v): A) Improvement on polyphenol content according to the extractions cycles (n = 5). B) TPC from each cycle and condition (n = 5). Lowercase letters means significant difference between polyphenol content on each cycle on the same condition and extraction type ($P < 0.05$). Capital letters means significant difference between the same cycles between the conditions of the same extraction type ($P < 0.05$). *Signalized cycles were executed only on the respective condition. I – FVF:ETOH (1g:15mL); II – FVF:Viscozyme®:ETOH (1g:25µL:15mL); III – FVF:PC:Viscozyme®:ETOH [(1g:1g):25µL:30mL].

Viscozyme® is an enzyme complex that includes cellulases, hemicellulases, and pectinases and is highly compatible with the FVF matrix (48.4% of the biopolymers are dietary fibre as cellulose, hemicellulose, soluble lignin, insoluble lignin and resistant starch) (12), demonstrating that this enzyme complex can be used to improve extraction of polyphenols (5). These results are in accordance to reports that demonstrate increase of extraction of polyphenols applying enzymatic treatments (5).

The HAE extraction (Figure 1.B) demonstrates a gradual decrease of the extraction capacity as the number of cycle's increases similar in all conditions, probably due to the

reduction of free polyphenols on FVF. However, on the 6th cycle the extraction had a significant increase in all conditions, compared with the 5th cycle. It could be related to the long exposure to 40 °C temperature, since heating may weaken the hydrogen bonds between polyphenols and polymers promoting the release of these molecules (2).

On UAE, eight cycles were necessary on condition I, and seven for conditions II and III (Figure 1.A). All conditions had similar extractions. However, the total phenolic content of the UAE.III was significantly lower than the others, possibly due to the presence of pineapple crown structural polymers. The pineapple crown cellulose, hemicellulose and lignin can act as a physical barrier for cavitation effect reducing the extraction efficiency (6).

The UAE conditions were similar (Figure 1.B). After the 2nd cycle, a significant reduction on the extraction capacity of each cycle was observed, followed by a gradual decrease on the extraction. This could be related to the high degree of cell disruption on the FVF, caused by cavitation on the 1st and 2nd cycles already, leading to a higher concentration of polyphenols extracted on these cycles (4). The extraction of condition III had different profile. UAE.III presents the 1st cycle extracting more efficiently than the UAE.I and UAE.II 1st cycles, but with a significant reduce of the capacity of the 2nd cycle. It may be related with the particle size of the pineapple crown applied on the FVF. The higher particle size reduces the surface area exposed to cavitation, decreasing its effect.

It is possible to observe that the cycles 1 to 3 are the most relevant cycles, extracting between 80% (HAE) to 90% (UAE) while the cycles 4 to 6 had gradual decreases on the extraction capacity.

Comparing the condition I with the condition II (Figure 1.A), the enzymatic treatment on HAE (>15%) was much more significant than UAE (<5%) in terms of increase of extraction capacity. However, when condition I is compared with condition III, the condition III had lower effect on TPC (UAE.III). It may be related with the enzyme concentration on this condition, since the enzyme:substrate ratio is a relevant parameter to observe on the effect of enzymatic treatments (5). The pineapple crown also have relevant proportions of cellulose, hemicellulose and lignin (20) that could act as a competitive substrate for Viscozyme®. Viscozyme® was kept as 25 μL g^{-1} of FVF on the condition II, but on the condition III the proportion of enzyme to total solids was diminished to half. This effect can reduce the potential of extraction (5). Also, other studies with Viscozyme® suggest the use of higher concentration of enzyme:substrate up

to 10% (v/w) (21).

In a previous study, the FVF presented total polyphenol content of 22.49 ± 1.59 mg GAE g^{-1} of flour (13), demonstrating that the antioxidant potential of the matrix can't be explored with one single extraction (cycle 1), with approximately 60-70% of the polyphenol remaining on the polymeric matrix of FVF (Table 1). On UAE, after one single extraction (cycle 1), almost 50% of the total polyphenol content remains bounded on the FVF matrix. However, this is a relevant finding, since it demonstrates that the FVF have a high content of antioxidant dietary fibres on the matrix, which represents approximately 48% of total mass of FVF (12).

Table 1: Total polyphenol content on FVF extracts from hydro alcoholic extraction [HAE - 75% (v/v)] and ultrasound-assisted extraction (UAE) on three different conditions (I, II, III) considering as the final cycle the first of three cycles that did not presented statistical difference ($P < 0.05$) on TPC amount (n = 5).

Sample	First cycle extracted polyphenols (mg GAE.g^{-1} FVF)	Last cycle extracted polyphenols (mg GAE.g^{-1} FVF)	Total cycles	Polyphenol increase
HAE.I	30.67 ± 0.52^{aA}	77.19 ± 0.93^{aA}	6	252%
HAE.II	32.28 ± 0.34^{aA}	92.32 ± 5.79^{bA}	7	286%
HAE.III	25.16 ± 0.83^{bA}	76.28 ± 2.17^{aA}	6	303%
UAE.I	66.10 ± 0.85^{aB}	127.86 ± 4.09^{aB}	6	193%
UAE.II	71.84 ± 0.60^{bB}	134.48 ± 1.66^{bB}	5	187%
UAE.III	74.71 ± 1.25^{cB}	113.02 ± 2.71^{cB}	5	151%

Lowercase letters means difference between the conditions on the same extraction type and cycle ($P < 0.05$). Capital letters means difference between the extraction types on the same condition and cycle ($P < 0.05$). I – FVF:ETOH (1g:15mL); II – FVF:Viscozyme®:ETOH (1g:25µL:15mL); III – FVF:PC:Viscozyme®:ETOH [(1g:1g):25µL:30mL]. FVF: fruit and vegetable residues flour; GAE: gallic acid equivalent.

Comparing the conditions I and II, it was possible to observe that the enzymatic treatment with Viscozyme® was efficient on HAE and irrelevant for UAE.

Comparing HAE SEM images with FVF native SEM images (Figure 2), it can be seen that even the ethanol medium at extraction temperature could actively modify the cell wall structures of FVF, increasing the bioactive extraction (3).

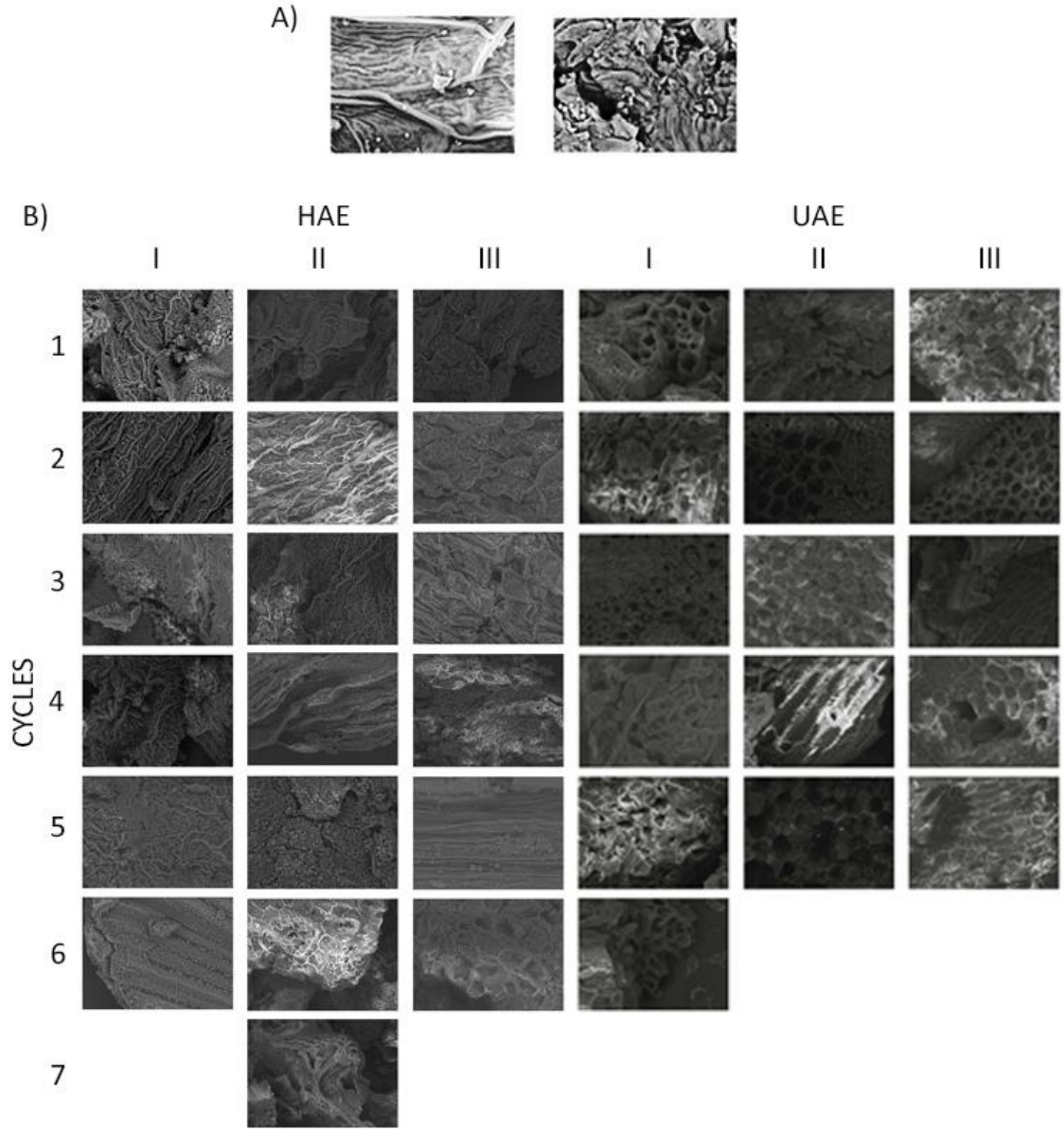

Figure 2: A) SEM images of FVF native microstructure. Magnifications at 1000x. B) SEM images from hydro alcoholic extraction [HAE -75% (v/v)] and ultrasound-assisted extraction [UAE] residues of three different conditions (I, II, III) at the final cycle. Magnifications at 1800x. I – FVF:ETOH (1g:15mL); II – FVF:Viscozyme®:ETOH (1g:25µL:15mL); III – FVF:PC:Viscozyme®:ETOH [(1g:1g):25µL:30mL].

Even if the FVF has a relevant protein content (10), the presence of natural bromelain may not promoted a significantly change on the microstructure of the FVF (Figure 2, Condition III), since all conditions presented similar SEM images. This is expected since cellulose, hemicellulose and lignin are the main structural polymers of FVF (12).

The UAE SEM images demonstrates great difference from the FVF native microstructure due the pores formed by the acoustic cavitation (4). The pores were formed on the 1st cycle already and remained on the samples until the final cycles. The condition III

microstructure were less affected by the UAE as expected due the protective effect of the pineapple crown against the cavitation´s effect (6).

Overall, the UAE was more efficient than the HAE on increasing polyphenols extraction from FVF (Table 2). The first cycle of UAE can extract approximately 215-300% more than 1 cycle of HAE, respecting the conditions. In addition, the best extraction condition was the UAE.I since it demands no enzymatic treatment and still had higher total polyphenol content than any HAE condition. However, HAE is simpler and cheaper, since only the extractor solution and a controlled heating equipment are required to perform it, while UAE requires specific equipment with sample cooling adjustment to be performed properly.

Table 2: Total polyphenol content on FVF extracts from the first cycle of hydro alcoholic extraction [HAE - 75% (v/v)] and ultrasound-assisted extraction (UAE) on three different conditions (I, II, III) (n = 5).

Sample Conditions	HAE TPC (mg GAE.g^{-1} FVF)	UAE TPC (mg GAE.g^{-1} FVF)	Difference between the extraction types
I	30.67 ± 0.52^{aA}	66.10 ± 0.85^{aB}	215,52%
II	32.28 ± 0.34^{aA}	71.84 ± 0.60^{bB}	222,55%
III	25.16 ± 0.83^{bA}	74.71 ± 1.25^{cB}	296,94%

Lowercase letters means difference between the conditions on the same extraction type ($P < 0.05$). Capital letters means difference between the extraction types on the same condition ($P < 0.05$). I – FVF:ETOH (1g:15mL); II – FVF:Viscozyme®:ETOH (1g:25µL:15mL); III – FVF:PC:Viscozyme®:ETOH [(1g:1g):25µL:30mL]. TPC: total polyphenol content; GAE: gallic acid equivalent

Although HAE is a simpler method of extraction, the FVF HAE extracts had a higher concentration of polyphenols (76.28 ± 2.17 to 92.32 ± 5.79 mgGAE.g^{-1}) than tomato peel extracts with 38.78 ± 0.05 mg GAE.g^{-1} (22) and wine shoot wastes with 32.1 ± 0.9 mg GAE.g^{-1} (23). The UAE extracts also had higher concentration of polyphenols (113.02 ± 2.71 to 134.48 ± 1.66 mg GAE.g^{-1}) than other ultrasound-extracts like: olive kernel with 60.75 ± 0.40 mg GAE.g^{-1} (24) and Persian lime (*Citrus latifolia*) wastes with 58.13 ± 0.4 mg GAE.g^{-1} (25). This demonstrates FVF as a polyphenol source since they could be easily extracted by simple and cheaper methods.

Encapsulation process

The powders obtained by the spray drying process can be used as food products ingredient since they are easier to be stored and added on food products formulation (17). Also, it is mentioned that the benefits of the use of maltodextrin as wall material for the food industry includes high water solubility, low viscosity and mild flavour, which are desirable for food

processing (9).

All conditions produced capsules with spherical shape, tipped morphology different sizes (Figure 3) and similar moisture contents, as expected from spray dried powders (1). In addition, no cracks were observed, indicating a good encapsulation process.

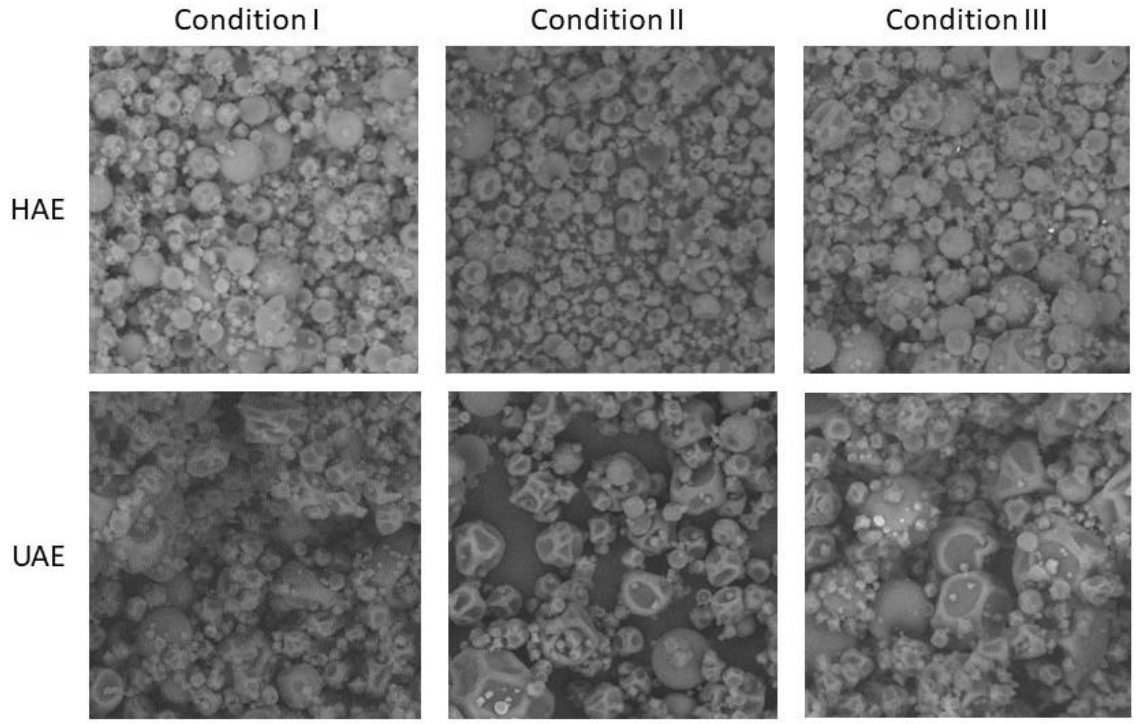

Figure 3: SEM images from spray-dried powders obtained from hydro alcoholic extraction [HAE -75% (v/v)] and ultrasound-assisted extraction (UAE) on three different conditions (I, II, III). Magnifications at1800x. I – FVF:ETOH (1g:15mL); II – FVF:Viscozyme®:ETOH (1g:25µL:15mL); III – FVF:PC:Viscozyme®:ETOH [(1g:1g):25µL:30mL].

Yield varied from 63.56% to 79.36% (Table 3), which was in accordance with other powders made with vegetable waste extracts as core and maltodextrin as wall material as the literature reports values as 72.81% to 76.49% for olive leaves extracts (26), 20.14% to 48.20% for grape pomace extracts (27). The difference on the yield values could be related with the extraction type and condition applied, since the drying parameters were maintained constant (17).

Table 3: Powder yield, polyphenol content and physical properties of spray dried powders obtained from hydro alcoholic extraction [HAE - 75% (v/v)] and ultrasound-assisted extraction (UAE) on three different conditions (I, II, III) (n = 3).

Parameters	HAE			UAE		
	I	II	III	I	II	III

Powder Yield (%)	79.36	68.89	78.00	78.91	63.56	72.63
Moisture (%)	5.57 ± 0.35^{aA}	6.5 ± 0.17^{bA}	5.67 ± 0.58^{aA}	5.20 ± 0.44^{aA}	4.47 ± 0.12^{bB}	4.63 ± 0.32^{bB}
Bulk density ($g.cm^3$)	0.257 ± 0.013^{aA}	0.304 ± 0.026^{bA}	0.300 ± 0.017^{bA}	0.401 ± 0.037^{aB}	0.381 ± 0.009^{aB}	0.367 ± 0.016^{bB}
Tapped Density ($g.cm^3$)	0.441 ± 0.003^{aA}	0.471 ± 0.005^{bA}	0.396 ± 0.004^{cA}	0.802 ± 0.073^{aB}	0.584 ± 0.025^{bB}	0.647 ± 0.046^{cB}
CI	41.62 ± 2.60^{aA}	35.32 ± 5.97^{bA}	24.22 ± 4.73^{cA}	47.16 ± 2.62^{aA}	34.81 ± 1.28^{bA}	43.21 ± 2.14^{aB}
CI classification	Low flowabillity	Low flowabillity	Intermediary flowabillity	Low flowabillity	Intermediary flowabillity	Low flowabillity
HC	1.72 ± 0.08^{aA}	1.55 ± 0.14^{bA}	1.32 ± 0.09^{bA}	1.90 ± 0.10^{aA}	1.53 ± 0.03^{bA}	1.76 ± 0.06^{aA}
HC classification	Highly cohesive	Highly cohesive	Intermediary cohesiveness	Highly cohesive	Highly cohesive	Highly cohesive
TPC (mg $GAE.g^{-1}$ powder)	1.00 ± 0.04^{aA}	1.23 ± 0.03^{bA}	0.54 ± 0.04^{cA}	1.50 ± 0.16^{aB}	1.92 ± 0.04^{bB}	0.62 ± 0.04^{cB}

Lowercase letters means difference between the conditions on the same extraction type ($P < 0.05$). Capital letters means difference between the extraction types on the same condition ($P < 0.05$). I – FVF:ETOH (1g:15mL); II – FVF:Viscozyme®:ETOH (1g:25µL:15mL); III – FVF:PC:Viscozyme®:ETOH [(1g:1g):25µL:30mL]. CI: Carr Index; HC: Hausner Correlation; TPC: total polyphenol content. CI: Carr Index; HC: Hausner Correlation; TPC: total polyphenol content; GAE: gallic acid equivalent.

The density is also an important factor to observe for storage. All conditions and treatments had intermediary to high cohesiveness as well as low to intermediary flowability. The differences between the bulk density and the tapped density reveals that capsules had low rate between the core material and the wall material (28).

The encapsulation process was proportional to the extraction efficiency from each extraction process and condition. The powder polyphenol content of the capsules obtained is in accordance with other powders obtained from vegetable wastes that were considered with high antioxidant activity, as a powder of citrus by-products conventional extracts that had 1.66 ± 0.02 mg $GAE.g^{-1}$ powder (29) and 1.69 ± 0.01 mg $GAE.g^{-1}$ powder for red pepper wastes conventional extract powder (30).

CONCLUSION

The best extraction method for FVF was the ultrasound-assisted extraction. The enzymatic process did not significantly influence this method, but it did on the hydro alcoholic extraction method increasing the extraction of polyphenols in approximately 15%. The use of the pineapple crown was not favourable for the extraction of polyphenols from FVF on both methods. Considering all steps included on each condition and extraction type, the best powder

obtained was UAE.I, since it has similar polyphenol content as the UAE.II with absence of enzymatic treatment and was obtained in much less time than the HAE powders. The results suggest that both FVF and FVF powders have potential as functional ingredient due to the high polyphenol content.

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CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest.

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