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Freeze-dried microencapsulation of bergamot pomace extract: stability and antioxidant performance in hydrophilic and lipophilic systems

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The use of microencapsulated antioxidant compounds is gaining increasing interest in both scientific and industrial fields due to their functional properties. In this study, a freeze-drying technique was applied to encapsulate an antioxidant extract obtained from bergamot pomace, a by-product resulting from the processing of this citrus fruit. Maltodextrin was used as a coating agent at a concentration of 20%. The resulting microencapsulated extract was then used to enrich two different matrices: (a) apple juice and (b) sunflower oil, in order to evaluate its effectiveness as a natural antioxidant. The antioxidant extract, the microencapsulated powder, and the enriched products were assessed for their physicochemical properties and antioxidant activity. In addition, the main characteristics of the enriched products were monitored during storage at 25 °C for 90 days. The freeze-drying process enabled the stabilization of the antioxidant compounds, facilitating their incorporation into both hydrophilic and lipophilic systems. The incorporation of the microencapsulated extract led to an increased content of polyphenols and enhanced antioxidant properties in both matrices, with these improvements maintained throughout the 90-day storage period.

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Sustainability spotlight

This study addresses current environmental sustainability challenges through the valorization of agro-industrial by-products. Specifically, it promotes the sustainable reuse of pomace derived from the processing of Bergamot, a citrus fruit typically cultivated in Calabria (Southern Italy). An antioxidant extract rich in bioactive compounds was obtained from this by-product and subsequently microencapsulated *via* freeze-drying. The encapsulated extract was used to enrich different food matrices (apple juice and sunflower oil), serving a dual function: as a natural preservative and to enhance antioxidant properties. The work aligns with a circular economy approach and supports several United Nations Sustainable Development Goals by promoting waste reduction, improving food quality, and fostering sustainable innovation.

1. Introduction

In recent years, consumers' nutritional needs have undergone significant changes. Awareness of the role of diet in maintaining health is steadily increasing, in parallel with the growing prevalence of chronic diseases linked to lifestyle and dietary habits. In this context, consumers are becoming more attentive to their nutrition, recognizing its direct influence on overall health.

At the same time, the rapidly growing global population has led to a higher demand for both food and energy, putting pressure on production systems to meet these needs. As a result, the number of food processing industries has significantly increased, leading to a substantial rise in food waste. This situation highlights the urgent need not only to prevent the generation of such waste, but more importantly, to develop effective strategies for its management and valorization. Among the innovations introduced by the food industry are the development of new food ingredients and materials, the adoption of advanced processing technologies, improvements in product quality, and the implementation of innovative packaging solutions.

However, research has increasingly focused on the potential of incorporating natural ingredients into food products to provide health benefits. These natural compounds reduced the need for potentially harmful synthetic additives, promoting the integration of bioactive natural substances as safe alternatives in response to market demand for innovative products.[1] In this contest, it emerges as a bridge between nutrition, health, and environmental sustainability. They combine both nutritional and functional properties while supporting sustainability

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through the incorporation of bioactive compounds recovered from agro-food by-products for the enrichment or fortification of new food products.[2–4]

The global functional food market is experiencing continuous growth, driven by the frequent introduction of new products aligned with current socio-demographic trends and widely accepted by consumers. This trend demonstrates significant potential, highlighting the need for ongoing research and development in this evolving field.[5,6]

Natural phenolic compounds, such as polyphenols, are particularly relevant in this context. These compounds are commonly extracted from plant matrices and agro-food waste and utilized in food production. However, their application is limited by challenges related to organoleptic properties, instability, and short shelf life. For instance, exposure to light, alkaline environments, and high temperatures affects their stability due to their structure, which contains multiple phenolic hydroxyl groups.[7]

A promising approach to overcoming these limitations involves the use of microencapsulation techniques, which enhance raw phenolic extracts derived from agro-food waste by protecting bioactive compounds from external environmental factors. This protection is achieved through a coating capsule that acts as a barrier, either *via* covalent or non-covalent binding of polyphenols to biopolymers. Several biopolymers can be used as wall materials, including gums, proteins, natural and modified polysaccharides, lipids and synthetic polymers (for example maltodextrins, chitosan, alginate, starch, whey protein, gum Arabic, xanthan gum, carboxymethylcellulose, *etc*). As a result, microencapsulation improves stability, bioavailability, and antioxidant activity of the encapsulated active ingredient.[8]

The interest in using microencapsulated bioactive compounds extends to both scientific and industrial fields,[9] and their functionality has been demonstrated in several studies.[10,11]

Hence, the valorization of natural extract through their use as novel natural ingredients is not only innovative but also ethical, given their high added value when recovered from agro-food waste. This type of application contributes to reducing environmental impact, optimizing natural resource utilization, and improving food product quality.

The primary objective of this research is to explore an alternative use for an antioxidant extract recovered by bergamot (*Citrus Bergamia* Risso) pomace. The extract was microencapsulated and incorporated into two different liquid systems: one hydrophilic (apple juice) and the other lipophilic (sunflower oil). The key properties of the enriched apple juice and sunflower oil were assessed over a 90-day storage period at 25 °C. For sunflower oil oxidative stability was also evaluated.

Therefore, this study contributes to the valorization of bergamot pomace, which has not yet been utilized in this manner, by developing a novel antioxidant ingredient and providing insights into its potential application in liquid food systems.

2. Materials and methods

2.1. Material and chemicals

Bergamot pomace (*Citrus Bergamia* Risso, BP) was supplied by an agro-processing company (citrus juices s.r.l.) located in the province of Reggio Calabria (Italy). Upon transport to the laboratory, BP underwent a drying process at 50 °C to reduce its moisture content to 12%, followed by an extraction process.

The preparation of the antioxidant extract from bergamot pomace (called BE) was carried out according to Gattuso *et al.*[12] with slight modifications to optimize the microencapsulation process. Briefly, 100 g of BP were mixed with 400 mL of an ethanol:water solution (1 : 1, v/v) and stirred for 30 min at 70 °C. The mixture was centrifuged (8000 rpm, 8 min, 4 °C), in a refrigerated centrifuge (NF 1200R, Nüve, Ankara, Turkey). The recovered liquid extract was concentrated using a rotary evaporator to remove ethanol, which was subsequently replaced with distilled water.

The preparation of the microencapsulated antioxidant extract (MBE) was performed following the method reported by Ballesteros *et al.*[13] with slight modifications. Maltodextrin (MD) was used as coating agent and added to BE at a concentration of 20%. The mixture was homogenized using an Ultra-turrax (T 25 digital, IKA, Staufen, Germany) at 7000 rpm until obtaining a homogeneous dispersion was achieved.

The prepared samples were frozen and subsequently freeze-dried using a VirTis lyophilizer (SPscientific, Gardiner, NY, USA) in a chamber set at −65 °C under a vacuum of 550 mtorr. The dried samples were then ground into a powder (referred to as MD20, maltodextrin 20%) and stored at room temperature in the dark, sealed containers until further analyses.

The obtained MD20 was used to enrich two different matrices: apple juice and sunflower oil. After preparation (Table 1), enriched samples (containing 2% of MD20) and control samples (without MD20) were stored in dark containers with minimal headspace and sealed. Each product was stored at 25 °C for the designated monitoring periods until further analysis.

2.2. Methods

2.2.1 Physicochemical characteristics of BE. Color parameters were determined using a Minolta CM-700d Spectrophotometer (PerkinElmer UV-vis λ2, Waltham, MA, USA). The L^*, a^*, b^* coordinates were analyzed and used to calculate Chroma (C^*) and hue angle (h°) according to the following equations:

$$C^* = (a^2 + b^2)^{1/2}$$

$$h^\circ = \arctan(b^*/a^*)$$

The pH was measured with a Crison pH-meter, basic model 20; while the total soluble solids (TSS) with a digital refractometer (PR-201a Atago).

Table 1 Composition of tested samples (%)

Sample	Liquid matrix (%)	MD20 (%)
Control juice (CJ)	Apple juice: 100	0
Enriched juice (EJ)	Apple juice: 98	2
Control oil (CO)	Sunflower oil: 100	0
Enriched oil (EO)	Sunflower oil: 98	2

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Total phenolic content (TPC) was determined according to the González–Molina *et al.*,[14] Briefly, 0.1 mL of BE, 5 mL of deionized water and 1 mL of Folin-Ciocalteu reagent were placed in a volumetric flask (25 mL) and mixed. After 8 min, 10 mL of Na_2CO_3, 20% were added. The reaction mixture was left to dark for two hours first to read the absorbance at 765 nm. The results were expressed as mg of gallic acid 100 mL^{-1} of extract (mg GAE 100 mL^{-1}).

The total flavonoid content (TFC) was determined according to Papoutsis *et al.*,[15] as follows: 500 μL of BE were added to 1000 μL of distilled water and 150 μL of $NaNO_2$ (5%, w/v) solution (in a total of 5 mL). The reaction mixture was left 6 min in dark and 150 μL of $AlCl_3$ (10%) solution were added. After 6 min, 2000 μL NaOH solution was added, and the volume was adjusted with distilled water. The absorbance was measured at 510 nm and the results were expressed as mg of catechin equivalents (CE) per 100 mL of BE (mg CE 100 mL^{-1}).

The total antioxidant activity (TAA): was measured through ABTS and DPPH assays.[12] For DPPH assay, 50 μL of BE and 2950 μL of DPPH (6×10^{-5} M) were mixed and after 30 min the absorbance was measured at 515 nm (against methanol as a blank). For the ABTS assay, 25 μL of BE and 2975 μL of $ABTS^{+}$ solution (7 mM) were mixed and after 6 min, the absorbance was measured at 734 nm against ethanol as a blank.

In order to calculate the TAA, results from both assays were expressed as mmol Trolox equivalents (TE) per Liter (mmol TE L^{-1}) of BE. The reduction in absorbance was plotted against Trolox concentrations ranging from 3 to 18 μM.

The identification and quantification of individual phenolic compounds (IPC) were performed following the method of De Bruno *et al.*[16] A 5 μL aliquot of each sample was injected into a UHPLC system (PLATINblue, Knauer, Berlin, Germany), equipped with a Knauer blue orchid C18 column (1.8 mm, 100 × 2 mm). The mobile phases consisted of (A) water acidified with acetic acid (pH 3.10) and (B) acetonitrile. The quantification of each phenolic compound was carried out using external standards, and results are expressed as mg L^{-1} of BE.

2.2.2 Physicochemical characterization of MD20. Water activity (a_w) and moisture content (MC%) were evaluated using a hygrometer (Aqualab LITE, Decagon, Nelson Court, Pullman, Washington and an Electronic Moisture Analyser (MA37, Sartorius, Goettingen, Germany), respectively.

The determination of bioactive compounds and the evaluation of the antioxidant activity of MD20 were performed following the procedure described by Zhang *et al.*[17]

To assess micro-encapsulation efficiency (M.E.%), total compounds in the microparticles (TCM) and compounds on the microparticle surface (CMS) were extracted. For CMS extraction, a mixture of ethanol/water (46.5/53.5, v/v) acidified with 2N hydrochloric acid (pH 2.00) was used, with a solvent to residue ratio of 8.7 mL g^{-1}. The mixture was homogenized using a vortex system and sonicated in an ultrasound bath for 50 min at 30 °C. It was then centrifuged (8000 rpm, 10 min, 4 °C) and filtered.

For TCM extraction, the same procedure as CMS was followed, but using only water as the extraction solvent.

The Folin– Ciocâlteu method, as reported by Azarpazhooh *et al.*,[18] was used to determine the TPC of TCM and CSM. Briefly, 0.1 mL of extract was mixed with 6 mL of water and 0.5 mL of Folin-Ciocâlteu reagent. After 8 min, 1.5 mL of Na_2CO_3 (20% w/v) was added, and the mixture was kept at room temperature in dark for 30 min. The absorbance was measured at 765 nm, and results were expressed as mg gallic acid equivalents (GAE) per 100 g of microencapsulate (mg GAE 100 g^{-1}).

TPC results of TCM and CSM were used to calculate the M.E., applying the formula below described by Rezende *et al.*:[19]

$$\text{M.E.\%} = [1 - (\text{CSM/TCM})] \times 100$$

Total flavonoid content (TFC) was determined as reported by Papoutsis *et al.*,[15] applying the same methodological procedure reported for BE (2.2.1). Results were expressed as mg catechin equivalent (CE) per 100 g of microencapsulated (mg CE 100 g^{-1}).

The ABTS and DPPH assays were determined according to procedure described by Sarabandi *et al.*[20] Results were reported as mmol Trolox equivalents per kg of microencapsulate (mmol TE 100 g^{-1}).

The identification and quantification of individual phenolic compounds (IPC) in MD20 were conducted on a 5 μL aliquot of the TCM extract using a UHPLC-DAD system, as previously reported for BE. Results were expressed as mg 100 g^{-1} of microencapsulated.

2.2.3 Physicochemical characteristics of apple juice enriched with MD20. Color parameters and pH of the juices were evaluated following the same methods reported for BE.

TPC was analyzed according to the method reported by Mafrica *et al.*,[21] and the results were expressed as milligrams of gallic acid equivalents per 100 mL of juice (mg GAE 100 mL^{-1}).

TFC, TAA and IPC were determined as described for BE.

2.2.4 Physicochemical characteristics of sunflower oil enriched with MD20. The color of sunflower oil samples was evaluated following the methods described for BE.

Total acidity (TA) was determined according to Official and Standards methods[22–24] and expressed as a percentage of oleic acid.

Peroxide value (PV) was assessed following the standard method of AOCS.[25]

Oxidative stability (OXITEST) was evaluated according to the method reported by Gattuso *et al.*[26] To determine oxidative stability, samples were subjected to accelerated oxidation conditions, and oxygen uptake was monitored in an Oxidation Test Reactor (VELP Scientifica, Usmate Velate, MB, Italy). Briefly, 5.00 g of sample were distributed in hermetically sealed titanium chambers, and pure oxygen was flushed into each chamber until a pressure of 6 bar was reached. The reactor temperature was set at 90 °C. The OXITEST measures the variation in absolute pressure within two chambers and automatically calculates the induction period (IP), expressed in hours, based on the inflection point of the pressure-time curve, following the AOCS International Standard Procedure.[27]

Regarding the antioxidant fraction of the sunflower oils, extraction was carried out according to the method of Baiano

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et al.[28] Briefly, 5 g of oil was mixed with 2 mL of methanol:water (70 : 30) and 2 mL of hexane. The hydroalcoholic phase was recovered and filtered. The spectrophotometric analysis of TPC was carried out following De Bruno *et al.*[3] The results were expressed as mg gallic acid equivalent per 100 g of oil (mg GAE 100 g^{-1}). TFC, TAA and IPC were evaluated as previously described for BE.

2.3 Statistical analyses

After calculating the mean and standard deviation of three measurements, the data were subjected to one-way analysis of variance (ANOVA), performed using SPSS software (Version 15.0, SPSS Inc., Chicago, IL, USA). Tukey's post hoc test was applied to determine significant differences between means at $p < 0.05$.

3. Results and discussion

3.1. Characterization of bergamot antioxidant extract (BE)

The analysis carried out on the BE enabled its characterization in terms of color, pH, total soluble solids (TSS), total phenolic compounds (TPC), total flavonoid content (TFC) and total antioxidant activity (TAA), as reported in Table 2.

The pH analysis confirmed the intrinsic acidity of bergamot pomace, with an average value of 3.3, which contributes to its microbiological stability by creating an unfavorable environment for microbial growth.

The evaluation of TPC, TFC and TAA in BE confirmed the notable presence of these compounds in bergamot, consistent with findings previously reported in the literature.[29,30]

Table 2 Physicochemical and antioxidant properties of BE

Color parameters	Results
L^*	42.39 ± 0.26
a^*	2.30 ± 0.02
b^*	3.63 ± 0.09
c^*	9.55 ± 0.37
$h°$	56.18 ± 0.62
Physicochemical and antioxidant properties	
pH	3.30 ± 0.05
TSS (°Brix)	6.05 ± 0.07
TPC (mg GAE 100 mL^{-1})	299.13 ± 1.56
TFC (mg CE 100 mL^{-1})	64.72 ± 1.01
ABTS (mmol TE L^{-1})	7.96 ± 0.45
DPPH (mmol TE L^{-1})	2.09 ± 0.15
Phenolic compounds (IPC) (mg 100 mL^{-1})	
p-Coumaric acid	5.77 ± 0.23
Ferulic acid	1.64 ± 0.19
Eriocitrin	6.31 ± 0.26
Neoeriocitrin	232.42 ± 5.26
Narirutin	3.10 ± 0.20
Naringin	280.05 ± 16.2
Neohesperidin	148.52 ± 2.39
Melitidin	52.16 ± 3.23
Brutieridin	107.45 ± 2.59

The main IPC quantified in the BE by UHPLC are also shown in Table 2. The results indicated that BE is a significant source of phenolic acids, particularly *p*-coumaric acid and ferulic acid, with concentrations of 5.77 ± 0.23 and 1.64 ± 0.19 mg 100 mL^{-1}, respectively. In agreement with earlier studies,[29–31] bergamot pomace was confirmed to be a rich source of flavonoids. The predominant compounds identified in the extract included eriocitrin, neoeriocitrin, narirutin, naringin, neohesperidin, melitidin, and brutieridin.

3.2. Characterization of the microcapsules

Table 3 reports the physicochemical analyses performed on the microencapsulated extract, including color, water activity (a_w), moisture content (MC), and antioxidant activity.

a_w and MC are key parameters affecting the shelf-life of microencapsulated powders. The MC value of 3.90 ± 0.21% was comparable to that reported by Sharifi *et al.*[32] In contrast, the a_w value of 0.18 ± 0.01 was lower than that reported by Díaz *et al.*[33] for microcapsules of blackberry juice obtained using maltodextrin as an encapsulating agent. The values observed in MD20 (a_w < 0.3 and MC <5%) suggest that the powder can be considered microbiologically and chemically stable over time.[34]

TPC was determined for both the total content in microcapsules (TCM) and the surface content (SCM), to calculate the microencapsulation efficiency (ME%), with results shown in

Table 3 Physicochemical findings of MD20[a]

Color parameters	Results
L^*	87.36 ± 0.17
a^*	1.99 ± 0.03
b^*	13.37 ± 0.08
C^*	81.56 ± 0.11
$h°$	91.37 ± 1.11
a_x	0.18 ± 0.01
Physicochemical and antioxidant properties	
MC (%)	3.90 ± 0.21
TCM (mg GAE 100 g^{-1})	947.35 ± 39.96
SCM (mg GAE 100 g^{-1})	28.99 ± 14.89
TFC (mg CE 100 g^{-1})	190.68 ± 14.89
ABTS (mmol Trolox 100 g^{-1})	11.14 ± 0.15
DPPH (mmol Trolox 100 g^{-1})	3.46 ± 0.21
E.E. (%)	94.28 ± 4.56
Phenolic compounds (IPC) (mg g^{-1})	
p-Coumaric acid	0.16 ± 0.00
Ferulic acid	0.03 ± 0.00
Eriocitrin	0.20 ± 0.00
Neoeriocitrin	7.32 ± 0.09
Narirutin	0.11 ± 0.01
Naringin	8.55 ± 0.10
Neohesperidin	4.75 ± 0.19
Melitidin	1.27 ± 0.08
Brutieridin	3.20 ± 0.11

[a] Color measurements revealed values of 87.36 ± 0.17 for L^*, 1.99 ± 0.03 for a^*, and 13.37 ± 0.08 for b^*, with a Chroma (C^*) of 81.56 ± 0.11. The hue angle ($h°$) indicated a color tendency toward red and yellow, with a value of 91.37 ± 1.11.

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Table 3. An ME% greater than 90% was observed, attributed to the low TPC detected in SCM (28.99 ± 14.89 mg 100 g^{-1}) and the high TPC in TCM (947.35 ± 39.96 mg 100 g^{-1}). This value is consistent with those reported by Saikia *et al.*[35] for micro-encapsulated extracts of *Averrhoa carambola* pomace using maltodextrin *via* lyophilization, which ranged between 78% and 97%.

Additionally, Table 3 reports the total flavonoid content (TFC) of MD20, with a value of 190.68 ± 14.89 mg 100 g^{-1}, as well as antioxidant activity assessed by ABTS (11.14 ± 0.15 mmol 100 g^{-1}) and DPPH (3.46 ± 0.15 mmol 100 g^{-1}) assays.

Chromatographic analysis of the TCM extract, performed using the UHPLC system, allowed the identification and quantification of the main phenolic compounds in MD20 (Table 3). Among them, the most abundant were the flavonoids naringin, neoeriocitrin, and neohesperidin, in agreement with the phenolic profile previously reported for the extract (Table 2).

3.3. Characterization of enriched apple juice with MD20

pH is an important parameter in food products, as it influences growth and can also affect the stability of many compounds in fruit juices.[36] In the analyzed samples, pH values (Fig. 1) showed a significant change in the control juice (CJ) stored at 25 °C, whereas no significant differences were observed in the enriched juice (EJ). Variations between samples stored at the same temperature were not significant until the 90th day. Specifically, for CJ, an increase in pH from 3.54 to 3.65 was observed during the storage period, while EJ maintained a stable pH throughout, with values ranging between 3.50 and 3.55.

Color is one of the primary quality attributes perceived by consumers and contributes significantly to the overall qualitative assessment of a product. Color changes observed during the storage of juice samples are reported in Table 4. The brightness (L^*) of both the CJ and the EJ showed statistical differences over time. No significant differences were observed between the samples until day 90, when a slight decrease was recorded. CJ did not show statistical differences in a^* value during storage. In contrast, EJ showed a different trend, with a positive value at T90, likely due to increased redness

Table 4 Color parameters of enriched apple juice[a]

Parameters	Time	CJ	EJ	Sign.
L^*	T0	49.5 ± 0.08^{a}	49.5 ± 0.06^{a}	ns
	T45	48.55 ± 0.56^{b}	49.31 ± 1.34^{a}	ns
	T90	47.4 ± 0.10^{c}	47.02 ± 0.03^{b}	**
	Sign.	**	**	
a^*	T0	−0.18 ± 0.01	−0.24 ± 0.02^{c}	**
	T45	−0.18 ± 0.03	−0.52 ± 0.19^{b}	**
	T90	−0.20 ± 0.07	0.07 ± 0.02^{a}	**
	Sign.	ns	**	
b^*	T0	1.25 ± 0.06^{c}	1.41 ± 0.03^{b}	**
	T45	1.63 ± 0.14^{b}	1.52 ± 0.19^{b}	**
	T90	1.90 ± 0.11^{a}	1.75 ± 0.17^{a}	**
	Sign.	**	**	
C^*	T0	0.80 ± 0.08^{c}	1.02 ± 0.04^{b}	**
	T45	1.06 ± 0.29^{b}	0.88 ± 0.06^{b}	ns
	T90	1.83 ± 0.20^{a}	1.55 ± 0.33^{a}	**
	Sign.	**	**	
h°	T0	98.34 ± 0.84^{a}	99.61 ± 0.92^{a}	ns
	T45	98.78 ± 1.26^{a}	98.07 ± 0.60^{b}	ns
	T90	96.20 ± 2.38^{b}	87.84 ± 0.79^{c}	**
	Sign.	**	**	

[a] Letters show significant differences as assessed by Tukey's post hoc test. Abbreviations: n.s: not significant; **: significance at $p < 0.01$; *: significance at $p < 0.05$.

associated with the presence of phenolic compounds. Both samples showed an increase in yellowness (b^*), with statistically significant differences over time and between samples, probably due to a browning effect. At T90, the b^* value was lower in EJ (1.75 ± 0.17) compared to CJ (1.90 ± 0.11).

Storage time also affected the C^*, which increased more markedly in CJ during storage, whereas EJ exhibited a more moderate change. The hue angle (h°), indicative of the color tone, showed a significant evolution over time for both samples. Initially, h° values were similar between the two juices with no statical differences. However, over time the color tended to shift towards more reddish tones in EJ with significant differences between samples.

The study of antioxidant activity and bioactive compounds allowed the release of phenolic compounds from the micro-encapsulated to the apple juice (Fig. 2). The statistical analysis

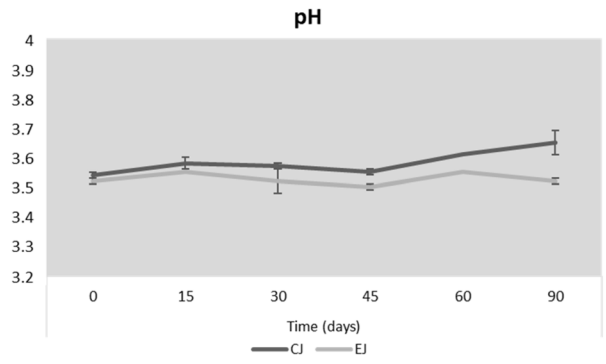

Fig. 1 pH evolution during storage of juice samples.

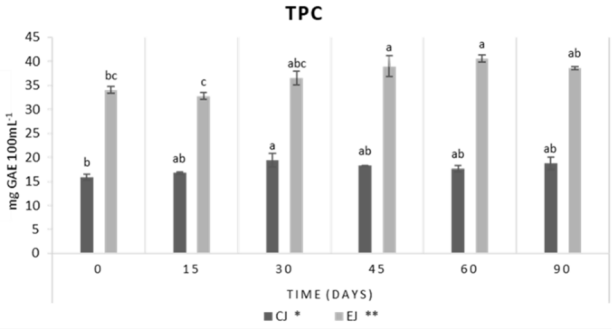

Fig. 2 TPC values of apples juice samples during storage time. Letters show significant differences as assessed by Tukey's post hoc test. Abbreviations: **: significance at $p < 0.01$; *: significance at $p < 0.05$.

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carried out on the different juice formulations (CJ and EJ) revealed statistically significant differences both between the control and enriched samples and within the individual samples during the 90-day storage period.

The TPC of CJ remained relatively stable over time, starting at 15.87 ± 0.60 mg 100 mL^{-1} on day 0 and reaching 18.73 ± 1.24 mg 100 mL^{-1} by day 90. In contrast, the EJ sample showed significant differences ($p < 0.01$). Specifically, EJ exhibited an initial TPC of 34.07 ± 0.68 mg 100 mL^{-1} at time 0, reaching a maximum value of 40.59 ± 0.58 mg 100 mL^{-1} at day 60. The increase in the phenolic content in EJ over time could be due to the storage temperature and the gradual release of bioactive compounds from the microencapsulated powder. The results obtained agreed with what reported by Hamid *et al.*[37] and Zokti *et al.*[38] who observed an increase in the total polyphenol content on beverages enriched respectively with microencapsulated phenolic extract of pomegranate flavedo and green tea extracts encapsulated in maltodextrin, gum arabic and chitosan.

Fig. 3 showed the changes in flavonoid content in apple juice samples during the storage period.

As observed TPC, statistically significant differences were also found for TFC, particularly in the EJ, which exhibited significantly higher values than the control juice (CJ), as well as a progressive increase in TFC during storage Specifically, EJ showed an increasing trend in TFC, starting at 127.54 ± 2.96 mg 100 mL^{-1} and reaching a maximum value of 214.32 ± 6.46 mg 100 mL^{-1} at day 60, which remained stable at day 90 (213.76 ± 2.74 mg 100 mL^{-1}). The observed increase in flavonoid content in EJ over time is likely due to the gradual release of bioactive compounds from the microencapsulated extract, which tend to diffuse into the hydrophilic matrix of the apple juice during storage.

TAA measured by ABTS and DPPH assays was illustrated in Fig. 4(a and b). As expected, EJ showed higher antioxidant activity than CJ for both the ABTS and the DPPH assay.

The ABTS values (Fig. 4a) showed greater antioxidant activity in EJ compared to CJ. EJ showed an increasing trend in antioxidant capacity, which correlated with the previously observed rise in phenolic compound content over time. In particular,

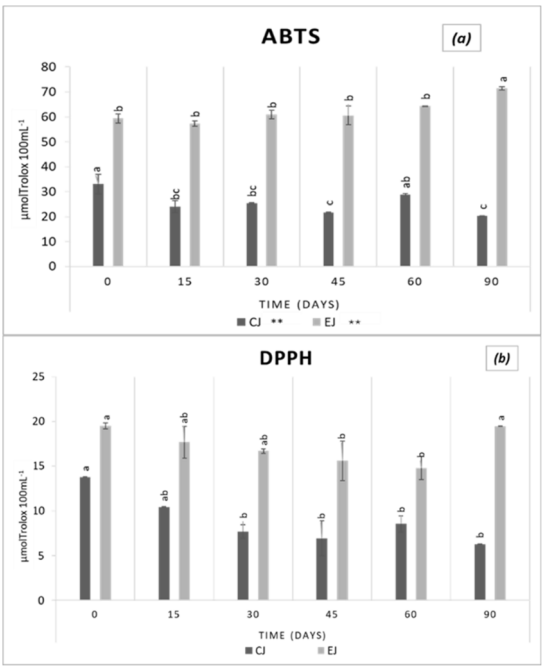

Fig. 4 TAA values of apples juice samples ((a): ABTS; (b): DPPH) during storage time. Letters show significant differences as assessed by Tukey's post hoc test. Abbreviations: **: significance at $p < 0.01$; *: significance at $p < 0.05$.

starting from the initial value, a progressive increase was recorded, reaching a maximum of 71.59 ± 0.64 μmol TE 100 mL^{-1} at day 90. In contrast, CJ showed an unstable and generally decreasing trend throughout the storage period.

The DPPH assay (Fig. 4b) revealed statistically significant differences in both CJ ($p < 0.01$) and EJ ($p < 0.05$). EJ maintained stable antioxidant activity over time, with values of 19.49 ± 0.01 μmol TE 100 mL^{-1} at day 0 and 19.46 ± 0.04 μmol TE 100 mL^{-1} at day 90. In the case of CJ, a marked decrease of approximately 50% was observed after 30 days, after which the value remained constant until the end of the storage period.

The chromatographic analysis of the main IPC detected in EJ by UHPLC was conducted at three points, day 0,45 and 90, to

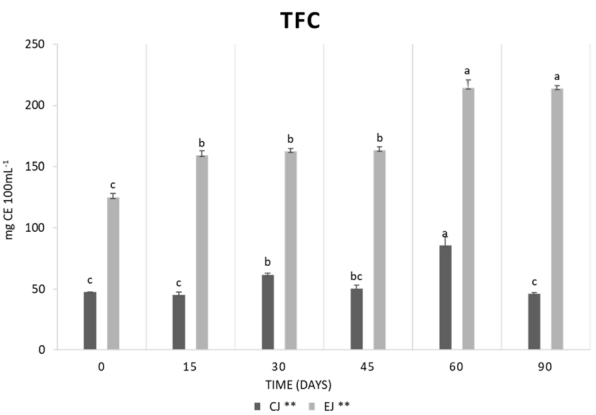

Fig. 3 TFC values of apples juice samples during storage time. Letters show significant differences as assessed by Tukey's post hoc test. Abbreviations: **: significance at $p < 0.01$.

Table 5 Individual phenolic compounds (IPC) determined in enriched apple juice (mg L^{-1}).[a]

IPC	T0	T45	T90	Sign.
p-Coumaric a.	1.68 ± 0.08	1.75 ± 0.3	1.6 ± 0.46	ns
Ferulic a.	n.d	0.19 ± 0.0	0.42 ± 0.27	ns
Eriocitrin	2.29 ± 0.1^{a}	2.31 ± 0.1^{a}	1.23 ± 0.29^{b}	*
Neoeriocitrin	88.18 ± 1.45^{b}	90.41 ± 0.28^{b}	107.16 ± 1.9^{a}	**
Narirutin	0.85 ± 0.04^{a}	0.87 ± 0.03^{a}	0.11 ± 0.01^{b}	**
Naringin	104.38 ± 1.47^{b}	111.54 ± 3.02^{b}	129.81 ± 0.10^{a}	**
Neohesperidin	57.44 ± 1.21^{b}	63.18 ± 1.93^{b}	94.47 ± 1.31^{a}	**
Melitidin	19.94 ± 0.56^{a}	17.18 ± 0.2^{b}	5.37 ± 0.31^{c}	**
Brutieridin	41.12 ± 0.42^{a}	41.23 ± 1.79^{a}	4.68 ± 0.08^{b}	**

[a] Letters show significant differences as assessed by Tukey's post hoc test. Abbreviations: n.s: not significant; **: significance at $p < 0.01$; *: significance at $p < 0.05$.

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track the behavior of the microencapsulated compounds in juice sample. As shown in Table 5, no statistically significant variations were observed for the phenolic acids (*p*-coumaric acid and ferulic acid). In contrast, flavonoid concentrations in EJ displayed statistically significant differences over time (Table 5). The major flavonoids: neoeriocitrin, naringin, and neohesperidin, exhibited a similar trend, with an increase in concentration observed after 90 days. The greater chemical stability of phenolic acids compared to flavonoids is attributed to their simpler molecular structure, which make them less susceptible to various types of degradation, including hydrolysis and oxidation. Specifically, phenolic acids contain fewer easily oxidizable functional groups and, unlike flavonoids, lack of catechol structure, which make them more stable and lower sensitive to oxidative and hydrolytic degradation.

Other flavonoids followed a different pattern. Specifically, eriocitrin, narirutin and brutieridin remained stable up to day 45, with concentrations comparable to those at time 0, followed by a significant reduction ($p < 0.01$) at day 90. Melitidin showed a gradual decrease, from 19.94 mg L^{-1} (T0) to 17.18 L^{-1} (T45), reaching 5.37 mg L^{-1} at T90. These different trends may be attributed to the variable release kinetics of individual compounds from the microencapsulated matrix. Nevertheless, the overall flavonoid content increased over time, suggesting a sustained release of phenolic compounds. This trend is influenced due to specific physico-chemical characteristics of the flavonoids, relating to their solubility, polarity, molecular size, encapsulation strength, hydrophilia and resistance to degradation which affect the rate of diffusion, and consequently on the availability of compounds during storage. A similar effect was reported by Wyspiańska *et al.*,[39] who observed a gradual increase in isoflavone levels resulting from the degradation of maltodextrin capsules and subsequent release of phenolic molecules into the solution.

3.4. Characterization of enriched sunflower oil with MD20 (EO)

The following results were obtained from the qualitative and quantitative analyses carried out on the enriched (EO) and control (CO) sunflower oil samples.

Color parameters of both samples are reported in Table 6. For all measured parameters (L^*, a^*, b^*, C^* and $h°$), statistically significant differences ($p < 0.01$) were observed over time.

For EO, L^* values decreased from 51.03 ± 0.12 at time 0 to 46.78 ± 0.07 at the end of the storage period. In contrast, a^* value increased, while b^* and C^* also increased over time, reaching values of 1.72 ± 0.04 (b^*) and 1.49 ± 0.07 (C^*) at day 90, indicating a greater tendency towards yellow. The hue angle ($h°$) increased after 15 days, followed by a gradual decrease, reaching the lowest value at the end of storage.

In control oil (CO), L^* also decreased by day 90. The a^* coordinate decreased until day 60, then slightly increased by day 90. As shown in Table 6, both b^* and C^* parameters followed a similar trend, decreasing until day 30 (with value of 0.50 ± 0.04 and 0.17 ± 0.02, respectively), indicating a temporary shift toward blue. However, during the final two monitoring points, both values increased again, suggesting a renewed tendency towards yellow.

Table 6 Color parameters of sunflower oil samples during storage time

Parameters	Time	CO	EO	Sign.
L^*	T0	51.06 ± 0.26^{a}	51.03 ± 0.12^{a}	ns
	T30	51.09 ± 0.03^{a}	49.80 ± 0.08^{b}	**
	T60	48.70 ± 0.02^{b}	46.60 ± 0.08^{d}	**
	T90	47.31 ± 0.03^{c}	46.78 ± 0.07^{c}	**
	Sign.	**	**	
a^*	T0	-0.31 ± 0.03^{b}	-0.31 ± 0.02^{c}	ns
	T30	-0.31 ± 0.03^{b}	-0.28 ± 0.02^{b}	ns
	T60	-0.61 ± 0.02^{a}	-0.38 ± 0.01^{d}	**
	T90	-0.27 ± 0.02^{a}	-0.19 ± 0.02^{a}	**
	Sign.	**	**	
b^*	T0	0.68 ± 0.08^{c}	0.79 ± 0.10^{c}	ns
	T30	0.50 ± 0.04^{d}	1.18 ± 0.02^{b}	**
	T60	1.72 ± 0.02^{b}	1.70 ± 0.03^{a}	ns
	T90	1.93 ± 0.03^{a}	1.72 ± 0.04^{a}	**
	Sign.	**	**	
C^*	T0	0.28 ± 0.05^{c}	0.36 ± 0.08^{c}	**
	T30	0.17 ± 0.02^{d}	0.73 ± 0.03^{b}	**
	T60	1.66 ± 0.04^{b}	1.52 ± 0.06^{a}	**
	T90	1.90 ± 0.06^{a}	1.49 ± 0.07^{a}	**
	Sign.	**	**	
$h°$	T0	114.80 ± 2.43^{b}	111.78 ± 2.27^{a}	ns
	T30	121.75 ± 3.30^{a}	103.19 ± 0.72^{b}	**
	T60	109.65 ± 0.53^{c}	102.76 ± 0.35^{b}	**
	T90	97.88 ± 0.59^{d}	96.42 ± 0.43^{c}	**
	Sign.	**	**	

Moreover, $h°$ values in CO changed significantly over the storage period, showing an initial that peaked at day 30 (121.75 ± 3.30), followed by a steady decrease up to day 90.

In edible oils, oxidation control is a key parameter that defines both quality and safety for human consumption. It is influenced by several intrinsic and extrinsic factors, and the enrichment with natural antioxidants represents an effective strategy for its preservation.[40] Total acidity value is a widely recognized indicator of oil quality. As clearly visible in Fig. 5a, the EO sample did not show significant changes in total acidity throughout the experimental period. In contrast, the CO sample showed statistically significant differences ($p < 0.05$), with a steady increase observed during the final two storage stages (T60 and T90). Considering the same storage conditions for both samples (CO and EO) its increase could be partially ascribed to the autoxidative process.[41] These findings support the conclusion that the addition of antioxidants in microencapsulated form is more effective than their incorporation as liquid extracts, as demonstrated by several studies.[42,43]

The graph reported in Fig. 5b displayed the increasing trend in the peroxide values in both oil samples, with high statistical significance ($p < 0.01$). Up to day 30, both samples remained relatively stable, displaying a slight upward trend, with peroxide values around 2.20 ± 0.42 meq O_2 kg^{-1} for CO and 1.85 ± 0.08 meq O_2 kg^{-1} for EO. From day 60 onward, the increase became more pronounced, culminating at day 90 with values of 9.99 ± 0.58 meq O_2 kg^{-1} for CO and 11.80 ± 0.42 meq O_2 kg^{-1} for EO.

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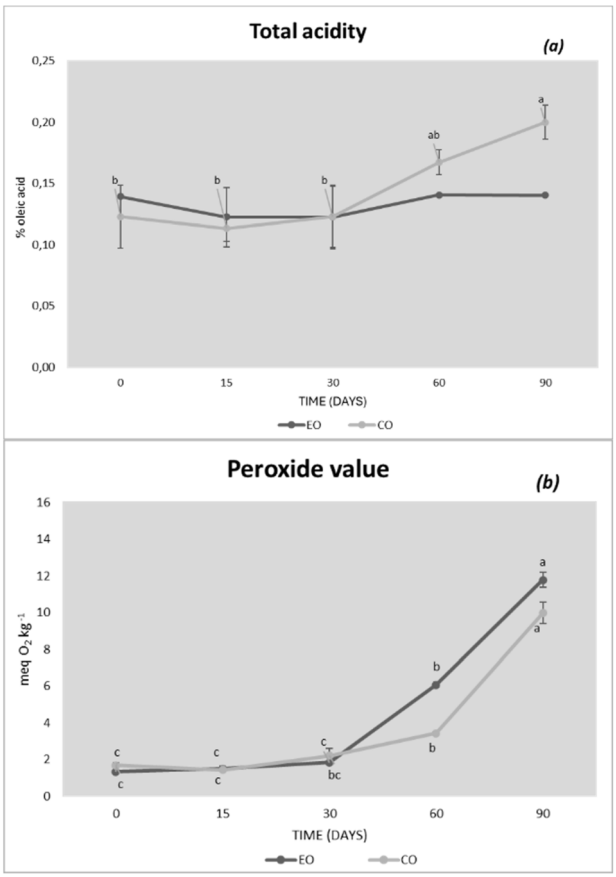

Fig. 5 Total acidity (a) and peroxide value (b) of sunflower oil samples. Letters show significant differences as assessed by Tukey's post hoc test.

Interestingly, the sample enriched with the microencapsulated extract (EO) exhibited a greater increase, which may reflect the complex interaction between antioxidants and the oxidative process at later storage stages. These results are consistent with those reported by Taghvaei *et al.*,[44] who observed an increase in peroxide values during 20 days of storage at 55 °C in soybean oil enriched with phenolic extracts from olive leaves encapsulated in maltodextrin, gum arabic, and a blend of both.

The oxidative stability of oil samples was studied by measuring the induction period (IP) using the Oxitest method. Fig. 6 illustrates the IP trend over time, represented by time-pressure curves at three monitoring points: 0, 45, and 90 days for EO. As expected, the presence of phenolic compounds added through MD20 resulted in a significant increase in IP after 45 days at 25 °C, reaching 14 : 12 (h : m) from an initial value of 13 : 16 (h : m). This improvement reflects the antioxidant effect of the bioactive compounds, in agreement with findings by De Bruno *et al.*[3] After 90 days, however, the IP of EO decreased compared to the value at T0, likely due to irreversible oxidation reactions occurring over time in combination with the storage temperature (25 °C), which can compromise the protective effect of the added antioxidants.

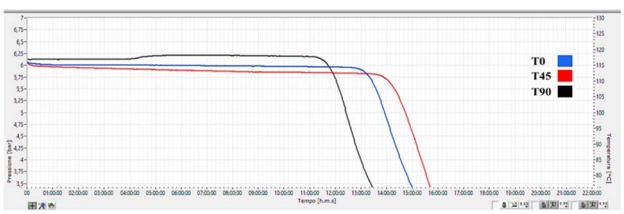

Fig. 6 Oxidation stability response on enriched oil.

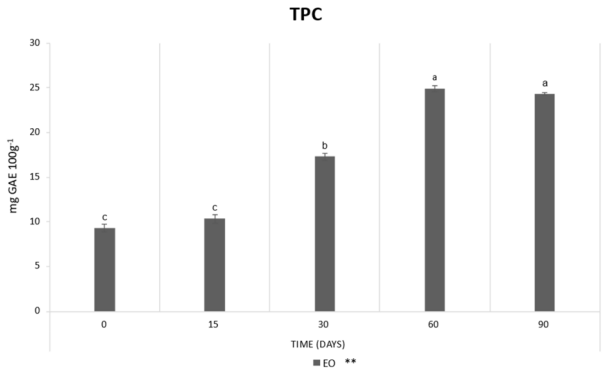

Fig. 7 Total phenolic compounds (mg GAE 100 g^{-1}) evaluated on EO during storage.

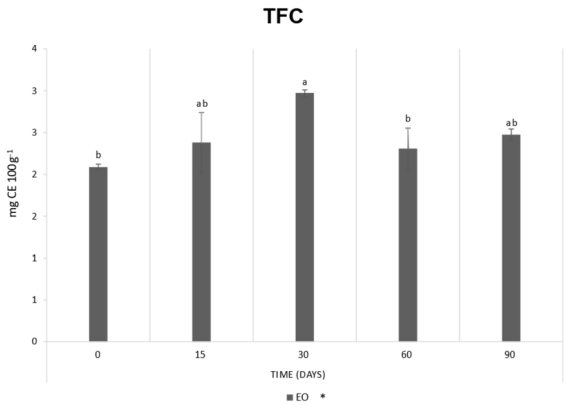

Fig. 8 Total flavonoids content (mg CE 100 g^{-1}) evaluated on EO during storage.

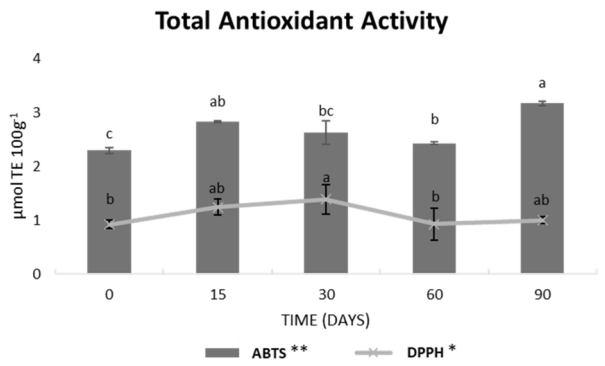

Fig. 9 Total antioxidant activity (µmol TE 100 g^{-1}) evaluated on EO during storage.

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Table 7 Individual phenolic compounds (IPC) determined in enriched sunflower oil (mg L^{-1})

IPC	T0	T45	T90	Sign.
p-Coumaric acid	0.7 ± 0.02b	1.01 ± 0.01a	0.98 ± 0.01a	**
Eriocitrin	1.03 ± 0.02b	1.27 ± 0.03a	1.13 ± 0.05 ab	*
Neoeriocitrin	37.77 ± 1.25b	46.71 ± 2.04a	43.31 ± 1.13 ab	*
Narirutin	0.97 ± 0.04b	1.28 ± 0.08a	0.39 ± 0.02c	**
Naringin	47.1 ± 0.66c	58.89 ± 0.4a	56.26 ± 0.65b	**
Neohesperidin	25.37 ± 0.62b	32.4 ± 1.61a	30.32 ± 0.14a	*
Melitidin	8.84 ± 0.03	10.42 ± 0	9.53 ± 1.41	n.s
Brutieridin	18.01 ± 0.79b	22.87 ± 0.77a	20.4 ± 0.48 ab	*

To evaluate the release and behavior of bioactive compounds from MD20 in sunflower oil, the antioxidant activity and capacity were assessed by analyzing total phenolic content (TPC), total flavonoid content (TFC), total antioxidant activity (TAA), and individual phenolic compounds (IPC).

The TPC values of EO, shown in Fig. 7, confirmed that enrichment with microencapsulated powder led to a measurable and statistically significant ($p < 0.01$) increase in phenolic content over time. The maximum value was observed at day 60 (207.91 ± 2.94 mg GAE 100 g^{-1}), which remained stable through day 90.

The TFC trend, illustrated in Fig. 8, showed a steady increase up to day 30, reaching a peak of 2.97 ± 0.04 mg CE 100 g^{-1}. Afterward, the flavonoid content slightly decreased and remained stable at days 60 and 90, with values around 2.50 mg CE 100 g^{-1}. The trends observed for both TPC and TFC are consistent with those reported by Taghvaei *et al.*,[44] who also noted a progressive release of both classes of compounds over time in oils enriched with microencapsulated phenolic extracts.

The antioxidant assays confirmed the antioxidant activity of enriched oil, attributable to the presence of phenolic compounds provided by the microencapsulated powder (Fig. 9). In particular, the ABTS assay showed a great antioxidant activity over time, with a minimum value of 15.70 ± 0.22 μmol TE 100 g^{-1} at time 0 and a maximum value of 20.43 ± 1.97 μmol TE 100 g^{-1} at time 90. Statistically significant differences ($p < 0.01$) were observed among the different time points.

The DPPH assay showed an increasing trend up to day 30, reaching a maximum value of 5.07 ± 0.99 μmol TE 100 g^{-1}. This was followed by a slight decrease at days 60 and 90. This trend is similar to what is observed in the analysis of TFC indicating that the antioxidant activity expressed by this *in vitro* assay is probably influenced by these compounds that act as hydrogen donors and radical scavengers with a corresponding fluctuation for both.

The behavior of MD20 in sunflower oil differed significantly from that observed in juice. As reported in Table 7, except for melitidin, differences of flavonoids content were found during the storage of EO. Eriocitrin, neoeriocitrin and brutieridin evidenced the same trend with a maximum level at 45th day and a subsequently slightly decrease ($p < 0.05$). Narirutin increased in the second monitoring time to decrease below the initial value at the end. High statistical differences ($p < 0.01$) were also found in naringin content which revealed an initial value of 47.1 mg L^{-1}, and higher values at time 45 (58.89 mg L^{-1}) and time 90 (56.26 mg L^{-1}). Moreover, *p*-coumaric acid and neohesperidin, after an initial increase maintained constant concentrations. The gradual release of encapsulated antioxidants into the oil phase is consistent with findings by Mohammadi *et al.*[45] The different behavior of MD20 in enriched juice (EJ) and enriched oil (EO) can likely be attributed to the hydrophilic nature of maltodextrin, the encapsulating agent. As maltodextrin is highly soluble in water, it facilitates a faster and more efficient release of the encapsulated compounds into aqueous matrices.[46] This could also explain the higher initial concentration of phenolic compounds found in juice samples compared to oil samples, where solubilization is slower due to the lipophilic environment.

4. Conclusions

The chemical characterization of the bergamot pomace extract confirmed its potential as a valuable source of polyphenols, highlighting the opportunity to enhance a citrus industry by-product through the extraction and valorization of its bioactive compounds. Microencapsulation of the extract proved effective in preserving its functional properties and enabled its application in both hydrophilic and lipophilic matrices, thus imparting nutraceutical and functional characteristics. Specifically, the use of 20% maltodextrin as an encapsulating agent allowed for the preservation of high polyphenol content, strong antioxidant activity, and excellent encapsulation efficiency.

The incorporation of the microencapsulated powder into a vegetable oil and an apple juice matrix led to a significant increase in polyphenol content and antioxidant capacity, which remained stable for up to 90 days of storage. The storage trial at 25 °C confirmed the oxidative stability of the enriched oil, while also highlighting the differential release behavior of the phenolic compounds in the two systems. These findings suggest that the encapsulation system performed as expected, supporting the preservation and gradual release of antioxidant compounds over time.

Enrichment with microencapsulated powder resulted in a significant enhancement of antioxidant properties in both food systems compared to the non-enriched controls. Overall, this study demonstrates a promising strategy for the functionalization of food products using natural micro-encapsulated ingredients derived from agri-food by-products, supporting

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a circular economic approach and sustainable innovation in the food industry.

Data availability

The data supporting this article have been included within the article.

Author contributions

Antonio Gattuso (A. G.) and Alessandra De Bruno (A. D. B.) conceived and designed the experimentation. A. G., Corinne Giacondino (C. G.) and Simone Santacaterina (S. S.) performed the experiments; A. G. and A. D. B. wrote the original manuscript; Amalia Piscopo (A. P.) validated and reviewed the final manuscript.

Conflicts of interest

There are no conflicts to declare.

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