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Bahtınur Taşcı
Ondokuz Mayis Universitesi
Saranya Selvaraj
saranyaselvaraj.tmp@sjp.ac.lk
University of Sri Jayewardenepura Faculty of Applied Sciences https://orcid.org/0009-0000-2146-2915
İlkay Koca
Ondokuz Mayis Universitesi

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Microencapsulation of red onion (Allium cepa L.) extracts by spray and freeze drying: Chemical characterization and release behavior during in vitro digestion

Bahtınur Taşcı[1]*, Saranya Selvaraj[2]*, İlkay Koca[3]

[1] *Department of Nutrition and Dietetics, Faculty of Health Sciences, Ondokuz Mayis University, Samsun, 55139, Turkey.*

[2] *Department of Chemistry, Faculty of Applied Sciences, University of Sri Jayewardenepura, Gangodawila, Nugegoda, Sri Lanka*

[3] *Department of Food Engineering, Faculty of Engineering, Ondokuz Mayis University, 55139, Samsun, Turkey.*

**Corresponding authors:*

Bahtınur Taşcı

e-mail: bahtinur.tasci@omu.edu.tr

ORCID: 0000-0003-3768-7363

Saranya Selavaraj

e-mail: saranyaselvaraj.tmp@sjp.ac.lk

ORCID: 0009-0000-2146-2915

Abstract

Red onions (*Allium cepa* L.) are rich in polyphenols, particularly quercetin. This study focused on the extraction, drying, and microencapsulation of red onion polyphenols via spray- and freeze-drying using maltodextrin (MD) and gum arabic (GA) as encapsulating agents. The extraction process was optimized using response surface methodology, with optimal conditions being 20 °C, 15 min, and 60% ethanol, yielding 1716.43 ± 57.44 mg/kg total phenolics, 349.5 ± 21.06 mg/kg flavonoids, and 29.16 ± 1.11 mg/kg anthocyanins. The obtained extracts were spray-dried, achieving the highest bioactive content with a 1:1 MD:GA ratio at 170 °C. Powdered extracts were microencapsulated using MD or MD:GA through both spray- and freeze-drying, and the resulting microcapsules were characterized to assess their physicochemical properties. Microencapsulated extracts retained over 90% of their bioactive content compared to the free extract and exhibited improved in vitro bioaccessibility of flavonoids, with notable resistance to degradation under simulated gastric conditions. Thermal stability tests confirmed that encapsulation significantly improved resistance to heat degradation, maintaining over 92% of polyphenols at 80 °C for 1 hour. In vitro digestion studies further revealed a controlled release of flavonoids, with minimal degradation in the gastric phase and enhanced bioaccessibility in the intestinal phase. Spray-drying with an MD:GA ratio of 1:1 at 170 °C was identified as the most effective and economical encapsulation method, suggesting its suitability for developing functional food formulations enriched with bioactive onion compounds.

Keywords: microencapsulation, red onion, *in vitro* bioaccessibility, phenolic compounds, flavonoids

1. Introduction

Allium species are found throughout temperate regions of the world, with a presence in Asia, Africa, Europe, and North America [1,2]. Among this genus, *Allium cepa* L. commonly known as onion, is used all over the world for its prominent nutritional, therapeutic and economic values [3]. Onion is commonly incorporated into a wide range of culinary preparations to enhance flavor and sensory attributes, as well as for its functional properties such as its antioxidant, anti-inflammatory, and antimicrobial properties due to their rich content of flavonoids (quercetin), anthocyanins, and sulfur compounds [4]; meanwhile they support cardiovascular health, blood sugar regulation, gut microbiota, and immune function, while also showing anticancer and neuroprotective effects [5] . Additionally, onions aid in wound healing, skin health, and serve as natural preservatives in food applications [6]. It is an important source of phytoconstituents containing predominantly flavonoids and organosulfur compounds [7]. The most dominant polyphenol in onions is quercetin which accounts for more than 60% of its polyphenolic content [8]. Like white and yellow onions, red varieties are rich sources of quercetin and kaempferol, primarily found as glucose-linked conjugates [9].

Previous studies have shown that onions have various biological activities relevant to human health [8]. Onions exhibit antimicrobial, antioxidant, analgesic, and anti-inflammatory properties, potentially aiding blood sugar management, cholesterol reduction, blood pressure regulation, and immune health [7]. Clinical evidence has suggested that onion consumption is linked to lower total body fat, especially visceral fat, alongside improvements in metabolic health indicators like triglycerides and C-peptide in overweight people [10]. Moreover, clinical results have shown that onions may help manage high blood sugar and insulin resistance in individuals suffering from cancer [11]. A clinical trial has also suggested that consuming raw red onion may hold promise for reducing cholesterol levels in individuals with polycystic ovary syndrome [12].

Due to its bioactive properties, onion is well-suited for use in functional foods designed to promote health. Nevertheless, the composition together with the biological activities of onion change during industrial food processing [6]. The chemical structure of polyphenols and phenolics, particularly flavonoids, makes them susceptible to changes during processing and storage, including oxidation, condensation, and degradation [13]. The use of polyphenolic compounds in different formulations is

limited as their stability is affected when subjected to light, temperature, or pH changes, which are factors encountered in food processing. Despite onions' well-documented health benefits, processing can lead to a loss or degradation of their beneficial phytochemicals. This has driven research into alternative methods for creating onion products that retain the stability of flavonoids, enhance their bioavailability, and extend shelf life.

Microencapsulation is a widely employed technique for preserving the phytochemicals and antioxidants of food products and supplements [14]. In microencapsulation, a coating agent surrounds small droplets or particles, forming microcapsules with a uniform or mixed structure. By creating a barrier against external factors, the coating materials minimize the core material's reactivity and safeguard it from degradation caused by oxygen, heat, moisture, and light [15].

Among the different microencapsulation techniques, spray-drying has become the predominant choice within the food industry due to its economic efficiency [16]. A liquid feed is atomized (broken into tiny droplets) using a hot gas stream, resulting in a powdered product. The core principle involves dissolving the core material in water to create a liquid emulsion. The resulting emulsion is then fed into a hot chamber (typically ranging from 100-300°C) where the water rapidly evaporates, leaving behind the encapsulated core material as a dry powder. On the other hand, freeze-drying is a microencapsulation method performed under low temperatures, making it ideal for preserving thermally sensitive bioactive compounds [16]. Freeze-drying removes water from a frozen emulsion by sublimation, concentrating the core material within a protective shell formed by the wall material. Nevertheless, it is associated with high energy costs and time in comparison to spray-drying [16].

Onion (poly)phenols and antioxidants are typically extracted from their natural source for microencapsulation applications. However, most studies lack optimization of the extraction process, leading to high starting material usage and potentially lower yields of these bioactive compounds. Optimizing the extraction method is crucial to minimize material requirements and maximize the recovery of (poly)phenols and antioxidants for a more efficient process. In fact, to date, the majority of the studies investigating microencapsulating onion extract have considered utilizing onion peel [17] and focusing on that sourced from yellow onion [18,19] for this purpose. Although utilizing plant waste is appreciated, yet most of the *in vivo* human clinical trials have reported the health benefits (such as anti-

diabetic, anti-obesity, and anti-hyperlipidemia) of onion 'vegetable' and not onion 'peel'. Additionally, previous studies have not investigated the effect of microencapsulation of onion extract on the bioaccessibility, bioavailability, and release behavior of (poly)phenols which creates another research gap in this area.

This study aimed to optimize the extraction process of red onions to maximize the yield of total phenolics, flavonoids, and anthocyanins. Once the optimal extraction conditions were determined, the extract was encapsulated using different methods. Microencapsulation was carried out using maltodextrin alone and a maltodextrin-gum arabic mixture, employing both spray-drying and freeze-drying techniques. The resulting microcapsules were then analyzed for their physicochemical properties, morphological characteristics, and thermal stability. Finally, an in vitro gastrointestinal digestion study was conducted to evaluate the release of onion flavonoids from the microcapsules, providing insights into their bioaccessibility and potential bioavailability.

2. Materials and methods

2.1. Materials

2.2.1. Chemicals and reagents

Maltodextrin (dextrose equivalent = 13-17), Folin–Ciocalteau reagent, quercetin, cyanidin-3-glucoside, pepsin (from porcine gastric mucosa), and solvents such as methanol, ethanol, and hydrocholoric acid (analytical grade) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Gum arabic, potassium chloride and sodium acetate were purchased from a commercial vendor (CARLO ERBA Reagents, Cornaredo, MI, Italy). Aluminium chloride, pancreatin, and bile extract porcine were purchased from Merck (St. Louis, MO, USA). Gallic acid and sodium carbonate were purchased from Riedel-de Haën, (Hanover, Germany).

2.1.2. Onion sample and preparation

125 The red onion samples were obtained from local producers in Tokat, Turkey in the Summer of 2018. On
126 the day of the experiment, the onions were peeled off, washed with distilled water and crushed with a
127 commercial blender (Waring Commercial Blender, USA).

128

129 ***2.2. Optimization of extraction process***

130 Response Surface Methodology (RSM) was employed to optimize extraction conditions using a three-
131 level Box-Behnken design implemented in Design Expert software (version 11.0, Stat-Ease, Inc.,
132 Minneapolis, MN, USA). The independent evaluated variables included ethanol-water ratio (%),
133 extraction temperature (°C), and extraction duration (min), with their influence on total phenolic content
134 (TPC), total flavonoid content (TFC), and total anthocyanin content (TAC) as response variables.
135 Optimum extraction conditions were determined according to the desirability function. A total of 17
136 experimental runs, including five center point replicates, were conducted across ethanol-water ratio (0-
137 80%), temperature (20-60 °C), and time (15-45 min) ranges. TPC, TFC, and TAC values were recorded
138 for each experimental condition. The experimental results were analyzed using a polynomial equation
139 in the form of Equation (1):

140 $$Y = \beta_0 + \sum_{n=1}^{3} \beta_i X_i + \sum_{i=-}^{3} \beta_{ii} X_{ii} + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i X_j \qquad (1)$$

141 where Y is the predicted response(s), X_i and X_j are the coded independent variables, β_{ij}, β_{ii}, and β_i are
142 the interactive, quadratic, and linear coefficients, respectively, and β is the model intercept.
143 For the extraction runs, 20 g of crushed onion and 100 mL of solvent was used. An ultrasonic bath
144 (Sonorex Super RK 510, Bandelin, Germany) with an ice battery (to maintain the temperature) was used
145 for the extraction at a power of 160 W and a frequency of 35 kHz.

146

147 ***2.3. Analysis of phenolic, flavonoid, and anthocyanin content***

148 The TPC, TFC, and TAC of the extracts were determined following the Folin-Ciocalteu method [20],
149 aluminum chloride method with minor modifications [21], and pH differential method [22],
150 respectively. Detailed descriptions of the methodologies are provided in the Supplementary File.

2.4. Encapsulation

The red onion extract obtained at optimum conditions was used to prepare the microcapsules. Prior to microencapsulation, the onion extract was concentrated with a rotary evaporator (Rotavapor® R-3, Büchi, Switzerland) operating under vacuum (23 mmHg) and a 40 °C water bath. The resulting extracts were combined with different wall material solutions at a ratio of 1:20 (w/w). The wall materials used were either pure maltodextrin (MD) or MD combined with different proportions of gum arabic (GA) as described below. The extract and the aqueous solutions of different coating materials were combined and mixed at 10,000 rpm for 10 min with the help of a blender (T18 digital ULTRA-TURRAX®, IKA-Werke GmbH and Co. KG, Staufen, Germany, UK). Finally, the samples were subject to spray-drying or freeze-drying as described below.

2.4.1 Spray-drying

To optimize the parameters for spray drying, a central composite design was employed within the framework of RSM. The variables considered were the inlet temperature (109.64-180.35°C) and the MD percentage (39.6-110.3%) while the responses considered included TPC, TFC, and TAC. Other operation conditions of the spray dryer (Mini Spray Dryer B-290, Büchi, Australia) were constant, including the maximum outlet temperature (90°C), feed rate (4 mL/min), air flow rate (38 m^3/h), atomization air flow rate (601 L/h), and solution temperature (60°C) [23]. Optimum extraction conditions were determined according to the desirability function. Following the optimization process, onion extracts were microencapsulated with (i) MD and (ii) MD and GA (1:1 w/w) to produce a spray-dried microencapsulated extract with maltodextrin (SDM) and spray-dried microencapsulated extract with maltodextrin and gum arabic (SDP), respectively.

2.4.2 Freeze-drying

Onion extracts were microencapsulated with MD and MD:GA (1:1 w/w) to produce a freeze-dried microencapsulated extract with maltodextrin (FDM) and a freeze-dried microencapsulated extract with maltodextrin and gum arabic (FDP), respectively. For the freeze-drying process, the mixtures of the

extract with coating material were initially frozen at -80°C for 24 h. Following that, the different samples were freeze-dried in a lyophilizer (FreeZone Plus 12 Liter Console Freeze Dryer, Labconco, USA) at 0.14 mbar and -85°C for 44 h [23]. The dried samples were reduced in size using a grinder.

2.4.3 Production yield of microcapsules

The production yield (PY) percentage of microcapsules was calculated based on the amount of starting material used in microencapsulation and the final based amount of microcapsules obtained according to Equation (2):

$$PY\ (\%) = \frac{Amount\ of\ microcapsules\ obtained}{Amount\ of\ starting\ material} \times 100 \qquad (2)$$

2.5. Analysis of microcapsules content and efficiency

The different types of microcapsules were analyzed for TPC, TFC, and TAC. A sample (0.1 g) of each microcapsule was weighed, dissolved in 1 mL distilled water, and then filtered through a 0.45 μm PTFE syringe for subsequent analysis. To determine the amount of surface phenolic content, 0.1 g of each sample was extracted using ethanol-methanol (1:1) [24]. After vortexing for 1 min, the mixture was filtered through a 0.45 μm PTFE syringe filter for analysis. The percentage of surface phenolic content (PSP) was then calculated using Equation (3). Finally, the PSP value was used to calculate the encapsulation efficiency (EE) by Equation (4). The surface phenolic content is an important aspect to consider when calculating the EE, that is because phenolic compounds present on the surface are not encapsulated and can be easily exposed to the external environment and undergo changes. Not considering the surface phenolics can overestimate the EE.

$$PSP\ (\%) = \frac{\text{Surface phenolic content}}{\text{Total phenolic content}} \times\ 100 \qquad (3)$$

$$EE\ (\%) = 100 - PSP \qquad (4)$$

2.6. Characterization of microcapsules

Microcapsules were characterized by particle size distribution [25], scanning electron microscopy for surface morphology analysis, infrared spectroscopy for surface functional groups , and differential scanning calorimetry for determination of glass transition temperature (T_g) following established methods reported previously [26].

2.7. Heat stability analysis

The heat stability of the extract and microcapsules (SDP and) was evaluated by incubating the samples in 20 and 80°C water baths for 60 min [17]. The amount of TPC and TFC before and after incubation was determined as previously described.

2.8. Release behavior during in vitro digestion

The release behavior of TFC of different microcapsules prepared by spray and freeze drying was investigated during *in vitro* digestion [27,28]. The changes in TFC of free extract was also investigated. TFC was selected (instead of TPC and TAC) as the most abundant polyphenol in onion is quercetin which is a flavonoid. Briefly, simulated gastric digestion was performed by adding 100 mg of each sample in 20 mL of an acidic solution containing pepsin (315 U/mL) at 37°C for 2 h. Subsequently, simulated intestinal digestion was conducted by dialyzing the gastric digest against a solution containing 1 M sodium bicarbonate, followed by the addition of pancreatin and bile and incubation at 37°C for 2 h. Samples from both gastric and intestinal phases were collected and stored at -20 °C for analysis. Detailed methodology is included in the Supplementary File. The bioaccessibility of flavonoids was calculated according to Equation (5).

$$\text{Bioaccessibility (\%)} = \left(\frac{\text{DV}}{\text{AV}}\right) \times 100 \qquad (5)$$

where DV is the amount of flavonoid content of microcapsules after gastric (GD) or intestinal digestion (IN), or the remaining phase from the small intestine digestion (OUT), and AV is the amount of TFC in the original sample [29].

2.9. Data analysis

The nonlinear regression analysis and 3D graphic construction were done using Design Expert software (Version 10.0, Stat-Ease Inc., USA). The effect of independent variables and their interactions on the

response was determined by ANOVA. The suitability of the designed models was calculated. The suitability of the designed models was calculated with the help of regression coefficient (R2), corrected regression coefficient (adj. R2), estimated regression coefficient (pred. R2) and standard deviation values. The data obtained for physicochemical properties and on powders were statistically analyzed with posthoc Duncan's test using SPSS software (Version 21, IBM, USA).

3. Results and discussion

3.1. Optimization of extraction conditions

3.1.1. Analysis of model

At this stage of the study, extraction conditions were optimized in terms of maximum total phenolic substance, antioxidant power, total anthocyanin and total flavonoid efficiency. Mathematical models showing the relationship between the independent variables in the optimization process and each response were created with the help of multiple linear regression analysis, and the models included linear, by adding quadratic and interaction effect terms, the increase in the sum of squares and model lag of fit values were examined. The statistical significance levels of these effect terms for the model were interpreted according to F and p values. While F value and p value express the importance of the model, a high F value indicates that the model is very important. Effects with a p value less than 0.05 were considered statistically significant in the regression analysis. The lack of fit value, that is, the model unsuitability value, shows the conformity of the designed model to the mathematical form. Statistically, the lack of fit of the mathematical form of the model should be insignificant ($p>0.05$) and important for the regression model.

The independent variables of optimization and the three responses are shown in **Table 3**. Responses were determined in ranges of 1225.71-1748.57, 101.24-324.22, and 10.82-47.89 mg/kg for TPC, TFC, and TAC, respectively. The lowest TPC and TFC were found at run 12 while the lowest TAC was found at run 11. The highest TPC was detected at run 10 while the greatest TFC and TAC were found at run 4. As shown, the values of the responses varied differently depending on the extractions, suggesting the importance of performing RSM to define the optimum conditions which give the maximum desired

responses. The regression coefficients of polynomial models were stepwise defined, and the reduced second-order models of coded units are shown as:

$$\text{TPC}\left(\frac{\text{mg}}{\text{kg}}\right) = 1863.16 + 9.07\text{X}_1 - 17.56X_2 - 8.27X_3 - 0.09X_1^2 + 0.15X_2^2 \quad (6)$$

$$\text{TFC}\left(\frac{\text{mg}}{\text{kg}}\right) = 78.10 + 5.22X_1 + 0.53X_2 - 0.04X_1^2 - 0.05X_3^2 \quad (7)$$

3.1.2. Effects of independent variables on responses

The statistical significance of the independent variables on the different responses is assessed through ANOVA, with results presented in **Table A (Supplementary File)**. The RSM model for TPC content was statistically significant ($p < 0.05$). TPC was mostly affected by the linear behavior of ethanol-water ratio (X1), temperature (X2) and time (X3). Figure 1 illustrates a significant increase in TPC as the ethanol concentration rose to 60%. However, further increases in ethanol concentration resulted in a decrease in TPC. Interestingly, neither temperature nor extraction time significantly affected the TPC within the tested range. In contrast to our findings, a study [30] observed that extraction time and temperature significantly influenced yield, with phenolic compound extraction efficiency increasing with temperature. The optimum extraction conditions were determined as 20°C, 15 minutes and 60% ethanol based on the desirability functions. However, they reported that as the temperature increases would adversely affect phenolic compounds because the properties of ultrasonic cavitation may change at high temperatures and phenolic compounds may oxidize. On the other hand, extraction time and ethanol concentration have been reported as determinant parameters for the recovery of TPC in several studies [31,32]. Kiassos et al. (2009) optimized phenolic compound extraction from onion waste using a central composite design, identifying both time and ethanol concentration as significant factors influencing total phenolic content recovery. While initially increasing phenolic yield, both parameters eventually reached an optimal point beyond which recovery declined [33]. Saikia et al. (2015) also observed a similar trend with temperature, finding an optimal extraction temperature of 40°C for phenolic compounds. However, ethanol concentration exerted a more pronounced effect on phenolic content than temperature, and their interaction positively influenced extraction efficiency [34]. Kaderides et al. (2015) also investigated the influence of temperature on ultrasonic-assisted extraction

of pomegranate peel phenolics, reporting an optimal temperature of 35°C. They reported that the vapor pressure of the solvent increased with increasing temperature and that the vapor pressure had a great effect on the formation and intensity of acoustic cavitation. They noted that more bubbles form at higher vapor pressure, but the bubbles collapse with less density because the pressure difference between the inside and outside of the bubbles is small. [35]. The underlying mechanism involves the disruption of plant cell walls by cavitation, enhancing mass transfer and the release of phenolic compounds into the solvent. While these studies align with the general trend of an initial positive impact of temperature on phenolic extraction followed by a decline, our results differed. These discrepancies likely arise from variations in raw materials, solvents, and extraction methodologies, as well as differences in the specific phenolic compounds present.

The model developed for TFC was found significant ($p < 0.0001$). The TFC of the purple onion extract was significantly and positively affected by linear terms of ethanol-water ratio (X1) and temperature (X2), and quadratic terms of ethanol-water ratio (X12) and extraction time (X32). The independent variables affected significantly the TFC ($p < 0.05$) where their increases occurred with the increase of TFC (**Figure 1**). The ethanol content exerted the most significant effect while the temperature and extraction time exhibited moderate effects. Similar to our results, previous studies have reported that an increase in the ethanol concentration [36] and extraction temperature [37] induced a high recovery of flavonoid compounds.

Ethanol concentration was a critical determinant of flavonoid yield in our onion extract, aligning with previous research. Jin et al. (2011) and Yang et al. (2010) observed enhanced flavonoid yields in quercetin and citrus flower extracts with higher ethanol concentrations. The improved solubility of flavonoids in solvents with similar polarity, such as ethanol, is likely responsible for this trend (Yang et al., 2010). While both ethanol concentration and extraction temperature significantly influenced flavonoid yield in our study, their interaction was not significant. Although Yang et al. (2010) reported an optimal extraction time of 50 min for flavonoids from citrus flowers, followed by a decline, our results did not exhibit a similar pattern. Both studies, including ours, confirmed the positive impact of temperature on flavonoid yield, attributed to increased molecular movement and subsequent enhanced solubility [38,39].

306 The anthocyanin content model was highly significant ($p < 0.0009$), demonstrating a strong correlation
307 with process parameters. Despite this, a significant lack of fit was observed, limiting the model's
308 predictive power for full optimization. Ethanol ratio, both linearly and quadratically, was the primary
309 factor influencing anthocyanin extraction. Although the overall yield was not significantly affected by
310 the independent variables, increasing them resulted in higher anthocyanin content (**Figure 1**).

311 *3.1.3. Multi-response*

312 In the optimization of ultrasound-assisted extraction conditions, the responses of the obtained extracts
313 to the amount of TPC and TFC and the relationship between the independent variables were found to be
314 significant, and these two responses fit the created model. Employing a desirability function, the analysis
315 determined the optimal extraction conditions to be: 60% ethanol concentration, a temperature of 20°C,
316 and a 15 min extraction time. The predicted values at this optimum point were 1742.65, 290.45, and
317 16.26 mg/kg for TPC, TFC, and TAC, respectively. For confirmation, additional experimental analyses
318 were performed, in triplicate, to determine the TPC, TFC, and TAC, under optimum conditions, which
319 yielded 1716.43 ±57.44, 349.53 ±21.06, and 29.16 ±1.11 mg/kg of TPC, TFC, and TAC, respectively.
320 Although these values seem to be close to predicted values, the experimental values of TFC and TAC
321 were found higher than the predicted values.

322

323 ***3.2. Optimization of encapsulation process by spray-drying***

324 Following optimization of the extraction process, the resulting extract was mixed with coating materials
325 and subsequently spray-dried. This encapsulation process was optimized using the central composite of
326 RSM and considering the air inlet temperature (120-170°C) and MD:GA ratio as independent variables.
327 The responses TPC, TFC, and TAC were maximized. The experimental results of all responses are
328 shown in **Table 4**. ANOVA and regression analysis were conducted to evaluate the model's fit, as
329 detailed in the Supplementary File (Table B).

330

331 *3.2.1. Effects of independent variables on TPC*

332 The results indicated that the combination of MD and GA as a coating yielded higher TPC than MD
333 alone (**Table 4**). The observed effective encapsulation is likely due to GA's highly branched

heteropolymer structure with covalently linked proteins, facilitating film formation with strong film-forming properties. (Kuck and Noreña, 2016). The model developed for TPC was significant ($p < 0.05$). The linear terms of temperature and MD ratio had a significant positive effect on TPC. The regression equation in terms of real variables for the TPC of the microcapsules was given as:

$$\text{TPC}\left(\frac{\text{mg}}{\text{kg}}\right) = 911.41 + 1.08X_1 - 19.78X_2 \quad (8)$$

As can be seen in **Figure 2(a)**, the TPC of microcapsules increased with the increase in temperature and MD:GA combination. However, the increase of TPC with MD:GA was observed when the MD ratio decreased, and the GA ratio increased. Several studies have reported that an increase in maltodextrin concentration caused a decrease in phenolic content yield [40,41]. The addition of GA to the wall material improved the TPC of microcapsules. Research has linked the GA coater's high phenolic substance efficiency in encapsulation to its molecular structure. This has been attributed to the fact that GA has a highly branched heteropolymer structure with a small amount of protein covalently bonded to the carbohydrate chain, allowing the formation of an effective encapsulate and functioning as an excellent film agent [42]. Since no study in the literature examines the antioxidant properties of purple onion encapsulates, the results were compared with similar studies on different raw materials. A previous study [43] found that açaí powders produced with GA in the spray dryer had the highest total phenolic content, which aligns with earlier findings [25]. MD and GA were used as coaters and it was reported that as the MD ratio increased, the phenolic substance yield decreased. The literature emphasizes that MD has a low film-forming ability, which affects encapsulate formation [44].

3.2.2. Effects of independent variables on TFC

The model developed for TFC was significant ($p < 0.05$). (**Table B – Supplementary File**). The linear and quadratic terms of temperature and MD:GA ratio had significant positive effects on the TFC. The regression equation in terms of real variables for TFC of the microcapsules was determined as:

$$\text{TFC}\left(\frac{\text{mg}}{\text{kg}}\right) = 235.98 - 1.92X_1 - 16.78X_2 + 6688020 \times 10^{-3}X_1^2 + 0.96X_2^2 \quad (9)$$

The TFC varied with the increase in temperature, with no clear trend. On the other hand, increasing the MD ratio, compared to GA, in the coating mixture, decreased TFC (**Figure 2(b)**). Thus, a higher GA proportion (and lower MD proportion), as coating material, positively affects the TFC. These findings are in agreement with several various studies where similar results have been reported [45,46]. Wu et al. (2014) reported that the MD:GA ratio and core:coater ratio had a significant effect on the encapsulation efficiency of flavonoid extract of *Rhodomyrtus tomentosa* (Ait.) Hassk. A study on the encapsulation of phenolic compounds extracted from sour cherry pulp reported that the coating efficiency of encapsulates with a core:coating ratio of 1:20 was higher than those with a core:coating ratio of 1:10, and increasing the GA ratio increased the coating efficiency [45].

3.2.3. Effects of independent variables on TAC

The model of TAC was not significant, indicating that the experimental data cannot be represented by the mathematical form of the model (**Table B – Supplementary File**). Although the effect of temperature and coating ratio on anthocyanin yield was not statistically significant, decreasing temperature and increasing GA ratio increased the TAC yield (**Figure 2(c)**). Similar results have been reported when optimizing the encapsulation conditions of grape anthocyanins [47]. The decrease in anthocyanin yield with increasing temperature could be explained by the degradation of anthocyanins at high temperatures [48,49].

3.2.4. Multi-response of optimization

The optimum conditions of the spray drying were determined by maximizing the responses. Based on the desirability function, the optimum spray-drying conditions were determined as 170°C and MD:GA ratio of 1:1. Under these optimum conditions, the predicted values were found as 995.70, 50.89, and 3.67 mg/kg for TPC, TFC, and TAC, respectively. For confirmation, additional analyses were performed in triplicate using the determined optimum conditions. The experimental values were determined as 998.35 ±3.64, 49.29 ±0.69, and 4.29 ±0.24 mg/kg for TPC, TFC, and TAC, respectively. Close agreement between the predicted and experimental values indicates good model validity, supporting the accomplishment and reliability of the optimization process.

In comparison to other studies, Jin et al. (2011), in their study in which they optimized different extraction methods for the extraction of quercetin from onion peel using the response surface method, determined that the optimum working conditions of the ultrasound-assisted extraction process at room temperature were 43.8% ethanol concentration and 21.7 min. Lim et al. (2007), in their study where they monitored the extraction properties of onion and the functional properties of the extracts with response surface methodology, obtained the maximum extraction efficiency as 61.77% ethanol concentration and 3.39 min extraction time [50]. Although the extraction methods used in these studies were different, results similar to our findings were obtained. In addition, similar results to our study were obtained in the study by Jeon et al. (2012) [51] in which they optimized flavonoid extraction from onion peel using the response surface method. As a result of the study, it was determined that flavonoid yield generally increased with the increase in ethanol concentration and temperature, which were selected as independent variables. In this study, it was determined that while ethanol concentration and temperature were significantly effective, their interactions were not significant.

Jin et al. (2011) found that in the optimization of ultrasound-assisted extraction conditions of onion peel quercetin with central composite design, ethanol concentration was effective on quercetin yield, while processing time was ineffective [38]. According to the results obtained by Jin et al. (2011), the effects of extraction temperature and ethanol concentration on flavonoid yield were found to be compatible with our study, while the effect of time on yield was found to be different.

A previous study [52] optimized anthocyanin extraction conditions from onion wastes using the response surface method, found the optimum extraction conditions to be 60% ethanol concentration, pH 2 and 3.7 hours. As a result of the study, the researcher reported that the highest anthocyanin yield was obtained at low pH and high ethanol concentration, and the most effective parameter on anthocyanin yield was time. Although the effect of the selected parameters on anthocyanin yield in our study was not statistically significant, it was determined that anthocyanin yield generally increased with increasing temperature, time and ethanol concentration, similar to the study of Makris (2010) [52].

3.3. Microcapsule content and formation properties

A total of four types of microcapsules were prepared for evaluation: SDM, SDP, FDM and FDP. Maltodextrin was selected as a wall material due to its good water solubility and low viscosity [53]. In addition, it is capable of stabilizing the core material (i.e., onion polyphenols) and regulating their release [54], thus contributing to an enhanced bioaccessibility. Nevertheless, MD has a low emulsifying ability [55], which could limit the use of MD-based microcapsules in different food matrices. Thus, in this study, the combination of MD and GA as a wall material was also investigated. GA has a good emulsifying ability [56] which can compensate for MD's low emulsifying ability.

The flavor retention, diversity, and low cost of carbohydrates such as starches, maltodextrins, and gums make these coaters a preferred option for encapsulation. Maltodextrins are tasteless, can be found in different molecular weights, show low viscosity in high dry matter and have matrix-forming ability. However, maltodextrins are generally preferred as auxiliary coating materials due to their low emulsifying properties and low capacity to retain volatile compounds [57]. Gum arabic is the most common hydrocolloid known for its emulsifying properties. Its emulsifying property is due to the protein structure contained in the gum, and its film-forming effect is due to the arabinogalactone fraction [58]. It has a wide use in encapsulation processes due to its high solubility, low viscosity, emulsification and film-forming properties and good retention of volatile compounds.

Gum arabic is generally preferred in the spray drying method due to its ability to emulsify and retain volatile compounds. Although a single encapsulation matrix does not have all the necessary properties, encapsulation properties can be improved by using mixtures of carbohydrates, proteins and polysaccharides in different proportions [59]. In our study, it is seen that in the MD:GA combination, the total amount of phenolic substances increases as the MD ratio decreases and the GA ratio increases.

The appearance of each type of microcapsule is shown in **Figure 3**. The amount of TPC, TFC, and TAC of each microcapsule was determined (**Table 5**). As can be seen, no significant difference was determined in the TPC and TFC values of the microcapsules obtained from freeze-drying and spray-drying using the same coating material. Similarly, a previous study found no significant difference in phenolic loss between spray-dried and freeze-dried grape skins encapsulated in GA and polydextrose

coaters [42]. However, in terms of the TAC of the microcapsules, significant differences were found between microcapsules prepared using different coating materials, as well as those prepared using the same coating material but with different drying techniques ($p < 0.05$). The freeze-dried samples had higher anthocyanin values than the spray-dried samples since the anthocyanins are quite heat-sensitive. Several studies have reported the effectiveness and efficiency of freeze-drying in protecting and preserving anthocyanins [60-62]. In both freeze-drying and spray-drying methods, the values of TPC, TFC and TAC of the microcapsules produced with MD:GA were higher than those of the microcapsules produced with MD. This finding indicated that the incorporation of GA into the coating mixture during the encapsulation process had a positive effect on the preservation of bioactive compounds. The PY and EE of the microcapsule formation are also shown in **Table 5**. The EE of onion phenolics was unaffected by variations in coating materials or encapsulation techniques ($p > 0.05$). However, freeze-dried capsules exhibited significantly higher percentage yields (PY) compared to spray-dried samples ($p < 0.05$). This discrepancy is attributed to material loss during spray drying, where feed components can become entrained in the hot gas stream and expelled from the system [63]. Similarly, Gabbay Alves et al. (2017), in their study using the response surface method in the microencapsulation of Theobroma cacao L. waste extract, found the optimum drying temperature to be 170 °C [64]. Similarly, Zhao et al. (2015) determined the optimum drying temperature to dry *Lycium barbarum* L. polysaccharide as 170 °C [65].

A previous study reported that the powders produced with GA in the spray dryer had the highest total phenolic substance content [43]. It has been emphasized in the literature that MD has a low film-forming ability and this affects encapsulate formation. In another study where MD and GA were used as coatings, it was reported that increasing maltodextrin concentration caused a decrease in phenolic substance yield [41].

3.4. Particle size analysis

The particle size of all the microcapsules produced in this study varied significantly ($p < 0.05$) (Table 6). The volumetric distribution of the FDP and SFP was more intense than the volumetric distribution of the FDM and SDM. Thus, the type of coating affects greatly the particle size distribution of the microcapsules. A study [17] has previously mentioned that the increase in the GA ratio increased the

particle size distribution in the microcapsules of onion skin. This is in line with our findings since the highest particle size distributions were obtained in FDP and SDP which had a coating material containing 50% of GA. The freeze-dried samples showed a denser volume distribution than the spray-dried samples (**Figure 4**), indicating that the freeze-dried microcapsules had larger particle sizes than the spray-dried microcapsules. The atomizer features used in the spray-drying, the low processing temperature applied in freeze-drying, the absence of a mechanical force to break up the frozen particles or change the surface structure during the drying and the agglomeration of the ice crystals during the freeze-drying are the main factors affecting the particle size of microcapsules [66,67]. In addition, the freeze-dried microcapsules exhibited larger particle size and inhomogeneous size distribution as they were broken down with the aid of a grinder. Similarly, a previous study [42] found that the D(4,3) value of freeze-dried microcapsules (104.3-684.9 μm) of grape skin was greater than spray-dried microcapsules (4.8-14.3 μm). It has been reported that the particle diameter of the spray-dried product varies between 0.5-50 μm [68], while that of freeze-dried products primarily depends on the grinding and powdering process following drying [69].

In the study, lyophilized samples show denser volumetric distribution than spray-dried samples. The fact that lyophilized encapsules have larger particle sizes than microcapsules obtained in the spray dryer is attributed to various reasons in the literature. The main reasons affecting the particle size are the characteristics of the atomizer used in the spray drying method, the low processing temperature applied in lyophilization, the absence of a mechanical force that will break the frozen particles or change the surface structure of the product during the drying phase, and the agglomeration that occurs in ice crystals during the freezing process [70]. In addition, since lyophilized encapsules are broken down with the help of a grinder, they exhibit larger particle sizes and a non-homogeneous size distribution.

3.5. Morphological features

SEM images of the microcapsules showed very different morphological properties depending on the drying techniques (**Figure 5**). Spray-drying yielded microcapsules with a generally spherical morphology, exhibiting surface depressions. The collapse or shrinkage observed in the microcapsules is

494 due to the formation of the precipitation of liquid droplets in the early stages of spray drying [71]. A
495 continuous and uniform surface structure, devoid of pores and cracks, indicates complete coverage by
496 the coating agent [72]. Freeze-drying resulted in microcapsules with an irregular morphology and a
497 fractured surface texture due to the grinding process. In general, several previous studies reported that
498 microcapsules obtained by spray-drying had a spherical structure, while those obtained by freeze-drying
499 had a sharp and broken glass structure [73,74]. In line with previous findings [75], spray-drying produced
500 red onion microcapsules with smooth surfaces. These findings showed that the drying techniques
501 affected the surface morphology of the red onion microcapsules. SEM analysis revealed smooth
502 microcapsule surfaces devoid of fissures, cracks, or voids, indicating successful encapsulation.
503
504 ***3.6. Glass transition temperature***
505 The T_g of a microencapsulated material serves as an indicator of its physical stability during extended
506 storage [76]. The thermograms of coaters and microcapsules are shown in **Figure 6**. The slope between
507 50-80°C is considered as the glass transition temperature of the microcapsules. Previous studies have
508 determined the T_g values of 51-60°C in the blackberry [77] or açai berry [78] microcapsules obtained
509 from MD and GA mixture. In consistence with our results, a previous study determined the T_g values of
510 45.83-52.38°C in the red onion microcapsules [79]. As can be seen from the thermograms, the DSC
511 thermograms and T_g values of the microcapsules and the coating materials displayed the same profile,
512 indicating that the core material adhered, and the encapsulation was effective.
513
514 ***3.7. Infrared (IR) spectroscopy analysis***
515 The spectra of the coating material and microcapsules are shown in **Figure 7**. Although the IR spectra
516 of the microcapsules were found to be similar, the extracts and microcapsules exhibited different peaks.
517 In the spectrum of MA, the peak between 3500-3000 cm^{-1} belongs to the OH bond. The peaks at 2993,
518 1149, and 1017 cm^{-1} were associated with C-H stretching, C-C bond, and C-O groups of carbohydrates,
519 respectively. These peaks presented the bonds found in the structure of MD [80]. For the spectrum of
520 GA, the peaks at 3600, 2990, 2962, 1693, 1413, and 1061 cm^{-1} belong to the N-H bond, aromatic CH_2,
521 CH_3 stretching, C=O (carbonyl) bond, C=C double bond and C-O bond, respectively [80]. The wide

peak between 3500-3000 cm^{-1} in the extract represents the characteristic OH stress peak. This peak corresponds to the OH stretching of H_2O and carbohydrates in the extract. The peaks at 3000 and 2942 cm^{-1} belong to the aromatic C-H bond, while the peaks at 1653, 1063, and 1412 cm^{-1} were related to C-C or C=C bond, C-O or C-O-C bond and -CH_2, -CH or =CH bond, respectively. These peaks correspond to the phenyl group present in phenolic compounds such as flavonoids [81]. The analysis of the FTIR spectra of the extract and microcapsules showed that the extract was effectively coated by the wall materials.

3.8. Thermal stability

The thermal stability of the TPC and TFC of the extract and microcapsules was evaluated at 20 and 80°C for 1 h (**Figure 8**). The extract exhibited the highest loss of TPC after 1 hour at 80 °C, while the microcapsules retained +92% of their content. Similarly, the extract exhibited the highest loss of TFC (59 ±3 %) when incubated at 80°C for 1 h, while the losses of less than 10% were determined for the microcapsules. These experimental results showed that the extract is more affected by external factors than microcapsules. Thus, the coating materials showed a protective barrier effect on the bioactive compounds, increasing the heat stability of the microcapsules. Many previous studies have reported the same tendencies for the encapsulated products where the coating materials have increased the heat stability of the microcapsules [82,83].

There are similar studies in literature to determine the heat stability of encapsulates. In a study in which phenolic compounds extracted from onion peel were encapsulated with the combination of MD-casein and MD-whey protein concentrate as coatings for heat stability analysis, microcapsules and uncoated phenolic powder were evaluated at 80°C for change in total phenolic compounds and antioxidant activity. While the total phenolic substance content of the uncoated phenolic powder showed a significant decrease of 45.88% in 1 hour at 80°C, there was no significant loss in the total phenolic content of the microcapsules. However, it was determined that MD-casein coating was more efficient than MD-whey protein concentrate coating material in protecting phenolic compounds during heat treatment [17]. In the study where *Malus floribunda* (ornamental apple) juice anthocyanins were encapsulated using (MD), (GA) and GA:MD (1:1) as coating materials, the thermal stability of the

550 encapsulated anthocyanins at different temperatures (70, 80 and 90 °C) was evaluated. It was determined
551 that the thermal stability of anthocyanins increased after the encapsulation process [84].

552 In the encapsulation of norbixin by spray drying method using MA and GA coaters, the heat stability
553 study was carried out at 60, 90 and 98 °C temperatures for 300 minutes in an aqueous model system.
554 Non-encapsulated norbixin samples were also evaluated for heat stability. As a result of the study, the
555 uncoated norbixin sample suffered a higher loss of bioactive compounds than the encapsulated sample,
556 revealing that microencapsulation improved the thermal stability of norbixin [83].

557

558 ***3.9. Release behavior of microcapsules during in vitro digestion***

559 An *in vitro* digestion model was employed to investigate the release profiles of the TFC of extract and
560 microcapsules throughout simulated gastrointestinal digestion. The release of flavonoid compounds
561 from the extract during gastric digestion (GD) was found to be significantly higher than the release of
562 flavonoid compounds from microcapsules ($p < 0.05$) (**Table 7**). In GD, the low release of flavonoid
563 compounds suggests the coating materials effectively acted as a barrier. This demonstrates the
564 encapsulation process's significant role in retaining these bioactive compounds.
565 Various studies have obtained similar results to our findings. A previous study [85] reported in their
566 study that microencapsulation of carob polyphenols showed a protective effect against pH changes and
567 enzymatic activities throughout digestion, thus contributing to increased bioaccessibility in the intestine
568 by promoting a controlled release and targeted delivery of the encapsulated compound. In a previous
569 study [45], results similar to the values we obtained were obtained. In the study where phenolic
570 compounds extracted from cherry pulp were encapsulated with MD:GA coaters at different ratios, it was
571 reported that microcapsules were absorbed to a higher extent in the small intestine than uncoated
572 phenolic powder, according to *in vitro* digestion test results. It was determined that the coaters used
573 protected the encapsulates from the stomach environment during gastric digestion and that the
574 encapsulation process was effective in retaining phenolic compounds. Additionally, the study found that
575 the MD:GA ratio used in encapsulation did not significantly affect bioavailability [45]. A previous study
576 [86] revealed that encapsulation of olive leaf extract provides preservation of olive oleoperin under

577 gastric conditions and controlled release under intestinal conditions, resulting in higher bioaccessibility
578 (58%) and potential bioavailability (20%).

579 In the IN medium, the release of flavonoid compounds from the microcapsules was found to be
580 considerably higher than in the GD medium (**Table 7**). While the release of flavonoid compounds in the
581 IN medium from the extract was found to be lower. In general, most of the microcapsules showed similar
582 behavior with no significant differences. It can be concluded that the release of flavonoid compounds
583 from microcapsules was low at the low pH of the GD medium, while higher in the IN medium. The
584 coating material which was damaged at the low pH in the GD medium, became easily soluble in the IN
585 medium, inducing a high absorption of flavonoid compounds. Therefore, the flavonoid compounds in
586 the microcapsule were effectively absorbed in the small intestine. This behavior could be explained by
587 the wettability at the high pH of IN medium and the solubility of the microcapsules. Data from **Table 7**
588 suggest incomplete absorption of TFC in the small intestine, with subsequent release of flavonoids in
589 the OUT medium. This is expected because, in the human body, the gut microbiota contributes to the
590 digestion of polyphenols by breaking them down into smaller phenolic compounds to be readily
591 absorbed [87,88].

592 In accordance with our results, a previous study [89] reported that the microencapsulated yellow onion
593 flavonoids were resistant to the gastric medium. Also, they found out that the maximum total flavonoid
594 release occurred at 90 min and concluded that the intestinal cells absorbed actively the bioactive
595 compounds at 90 min. In contrast, a different study [34] reported the highest phenolic release in the
596 simulated gastric medium with pH 1.2 compared to the simulated intestinal fluid with pH 6.8. The
597 observed variation in microcapsule behavior within the simulated digestive medium highlights the
598 dependence on several factors. These include the inherent properties of the coating materials
599 (composition, enzyme resistance/sensitivity), and the surrounding digestive environment (e.g., pH).
600

601 **4. Conclusions**

602 This study successfully encapsulated red onion extract using both spray-drying and freeze-drying with
603 MD and GA as coating materials. Prior to encapsulation, optimal ultrasound-assisted extraction

conditions (20°C, 15 min, and 60% ethanol) were established to maximize TPC, TFC, and TAC in the extract. Further optimization identified a 1:1 MD:GA ratio and 170°C in temperature as the best spray-drying conditions for microencapsulation. Importantly, both drying methods yielded microcapsules with similar TPC and TFC content, indicating effective retention of these bioactives. Characterization studies confirmed successful encapsulation, with spray-dried microcapsules exhibiting smoother, spherical morphology and freeze-dried samples displaying irregular, fractured surfaces. Heat stability tests confirmed the protective effect of the coating materials, enhancing the thermal stability of the encapsulated extract. Finally, *in vitro* digestion analysis demonstrated controlled release, with minimal bioactive release in the simulated gastric medium and a higher release in the intestinal medium, suggesting potential benefits for targeted delivery. Nevertheless, a portion of the flavonoid content remains unabsorbed, which suggests that future studies could consider adding *in vitro* microbial fermentation steps following *in vitro* gastrointestinal digestion to evaluate the role of gut microbiota in this process. Future studies could investigate incorporating onion microcapsules in food products and assessing the functional properties of the product.

Clinical trial number: Not applicable.

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Authors contribution: BT: conceptualization, methodology, software, data curation, investigation, formal analysis, and writing — original draft. SS: writing — original draft, and writing — review and editing. IK: conceptualization, project administration, and supervision.

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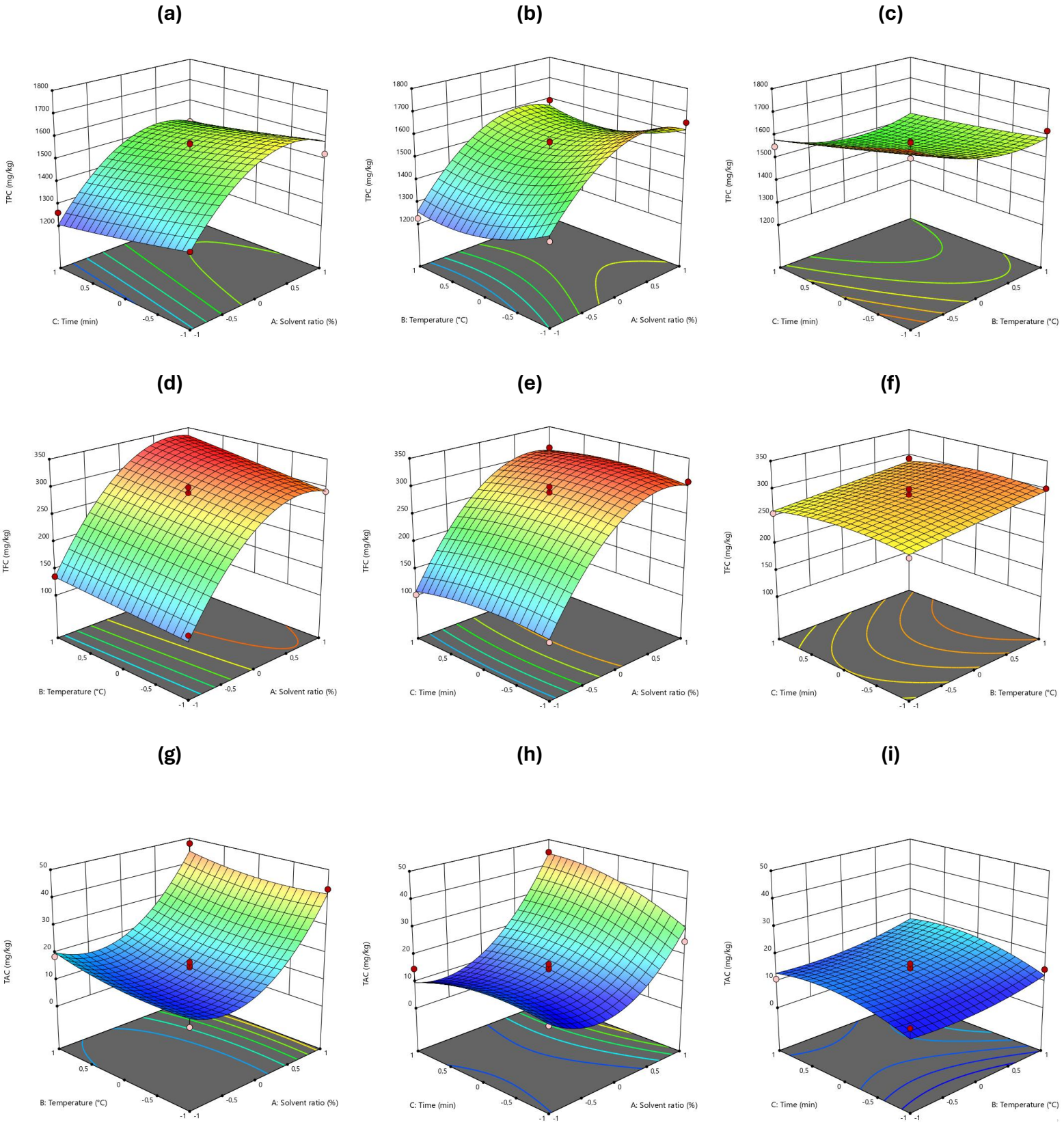
Figures



930

931 **Figure 1.** Estimated response surface by plotting total phenolic content (TPC) versa (a) solvent
932 ratio and temperature; (b) solvent ratio and time and (c) temperature and time; total flavonoid
933 content (TFC) versa (d) solvent ratio and temperature; (e) solvent ratio and time and (f)

temperature and time; and total anthocyanin content (TAC) versa (g) solvent ratio and temperature; (h) solvent ratio and time and (i) temperature and time. The independent variables of optimization were extraction temperature, ethanol-water extracting solvent ratio, and extraction time. The responses (dependent variables) were TPC, TFC, and TAC. The solid-to-liquid ratio was controlled (20 g of solid material with 100 mL of solvent; 1 g/5 mL).

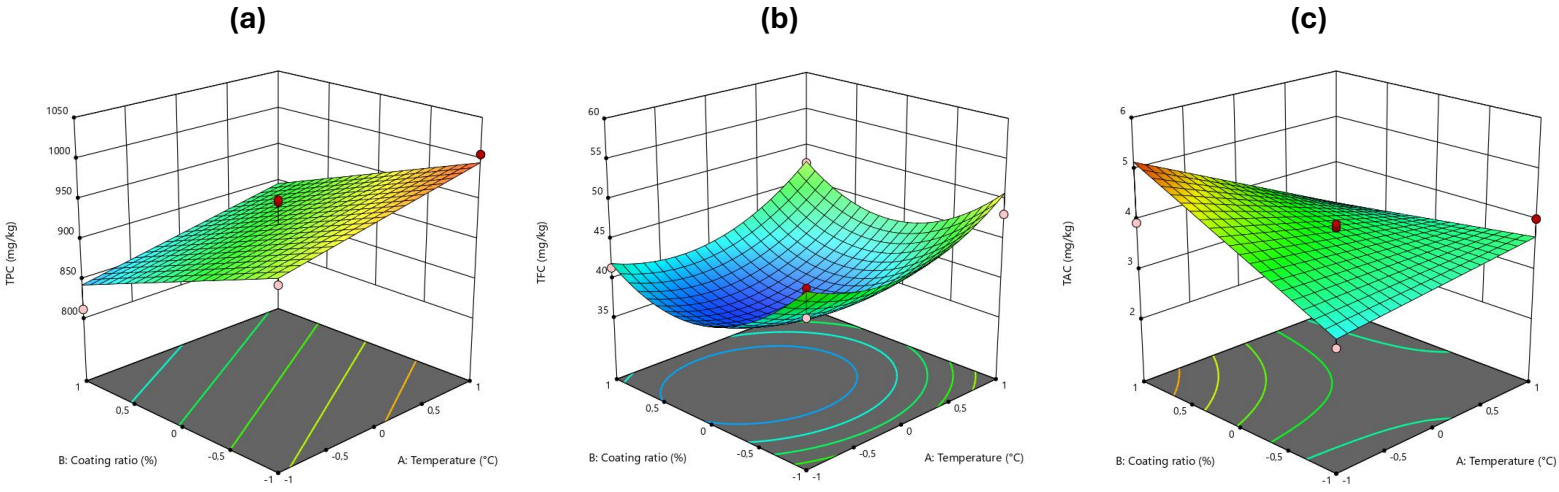
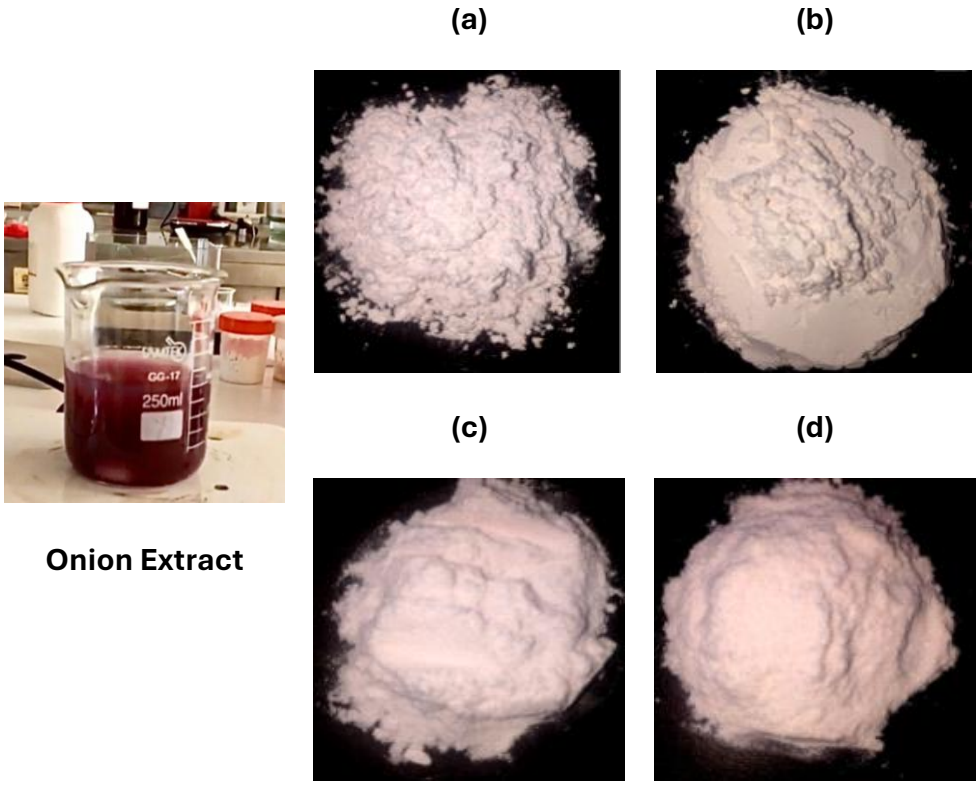


Figure 2. Estimated response surface by plotting (a) total phenolic content (TPC), (b) total flavonoid content (TFC), and (c) total anthocyanin content (TAC) versus inlet temperature and coating ratio. The independent variables were the inlet temperature of the spray dryer and the coating ratio. The responses (dependent variables) were TPC, TFC, and TAC.



951

952 **Figure 3.** Appearance of onion extract (left) and microencapsulated onion extract with (a)
953 maltodextrin, (b) maltodextrin and gum arabic using spray drying, and microencapsulated
954 onion extract with (c) maltodextrin, and (d) maltodextrin and gum arabic using freeze-drying.

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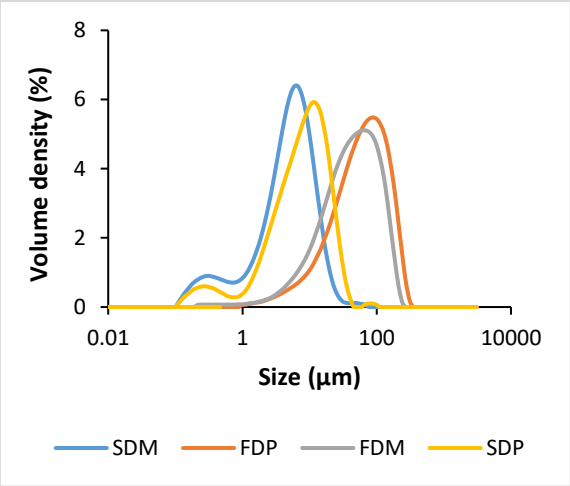

957

958 **Figure 4.** Particle size distribution of microencapsulated onion extract with maltodextrin
959 (SDM), maltodextrin and gum arabic (SDP) using spray drying, and microencapsulated onion
960 extract with maltodextrin (FDM), and maltodextrin and gum arabic using freeze-drying.

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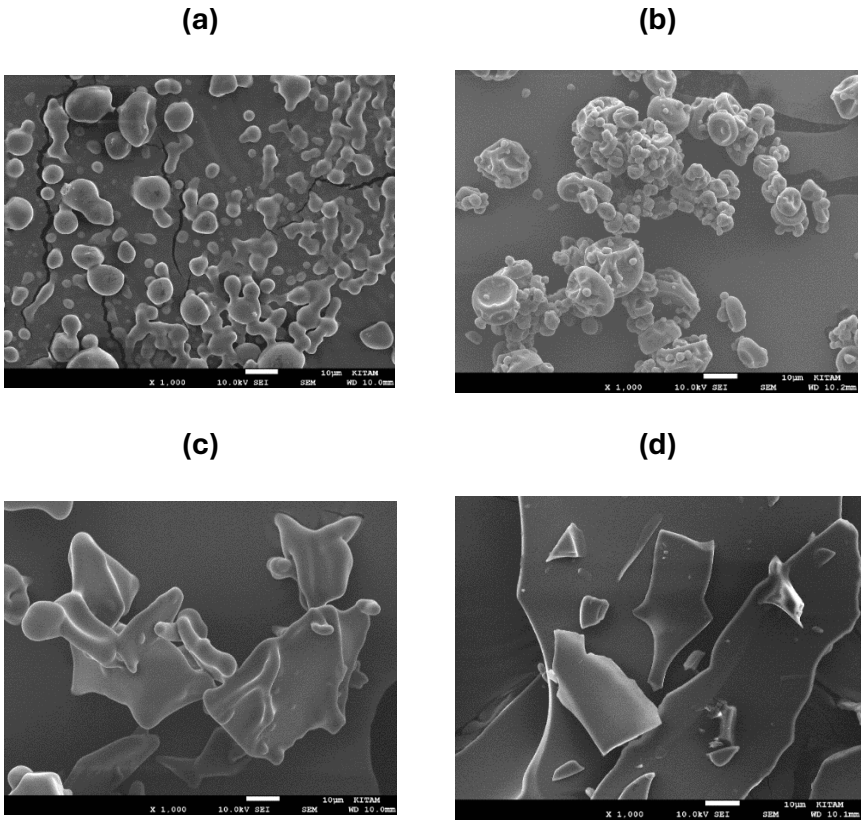
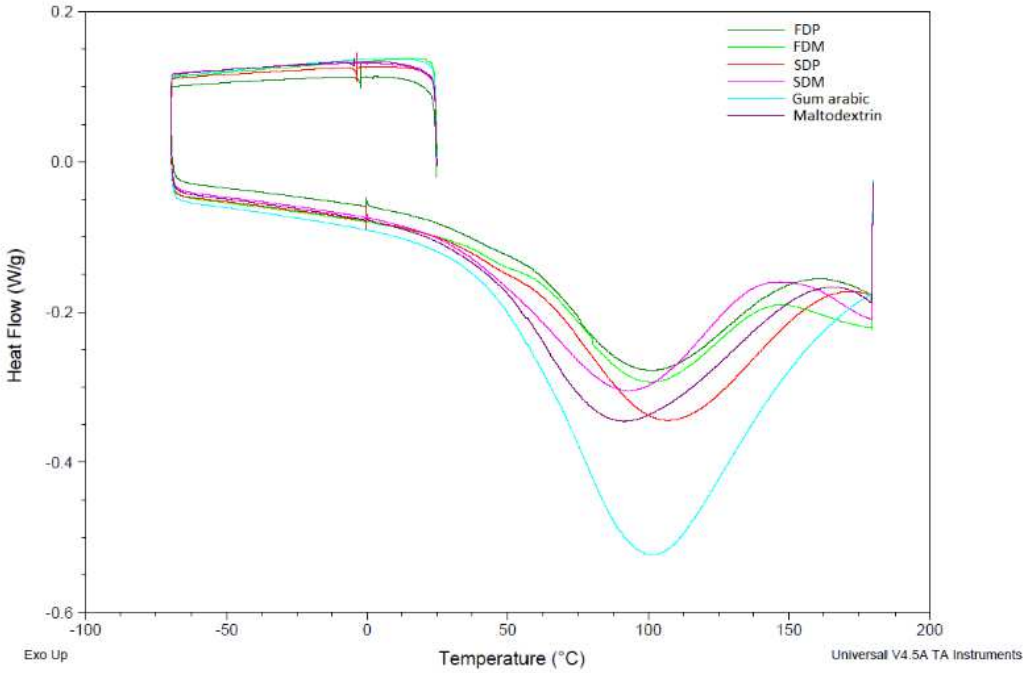


963

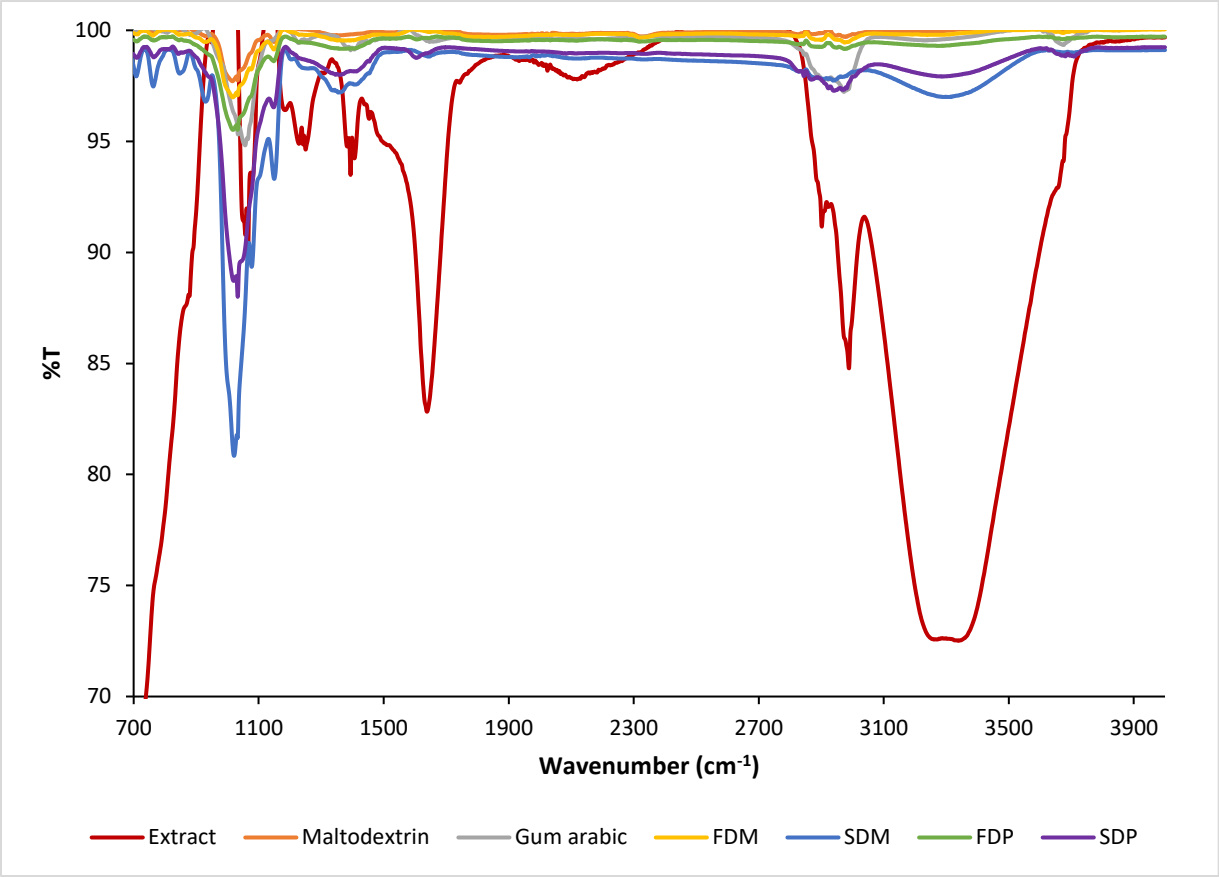
964 **Figure 5.** Scanning electron microscopy images of microencapsulated onion extract with (a)
965 maltodextrin X1000, (b) maltodextrin and gum arabic using spray drying X1000, and
966 microencapsulated onion extract with (c) maltodextrin X1000, and (d) maltodextrin and gum
967 arabic using freeze-drying X1000.



968

969 **Figure 6.** Thermograms of coating materials and microcapsules.

970



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972 **Figure 7.** Infrared spectrum of extract, coating materials, and microcapsules.

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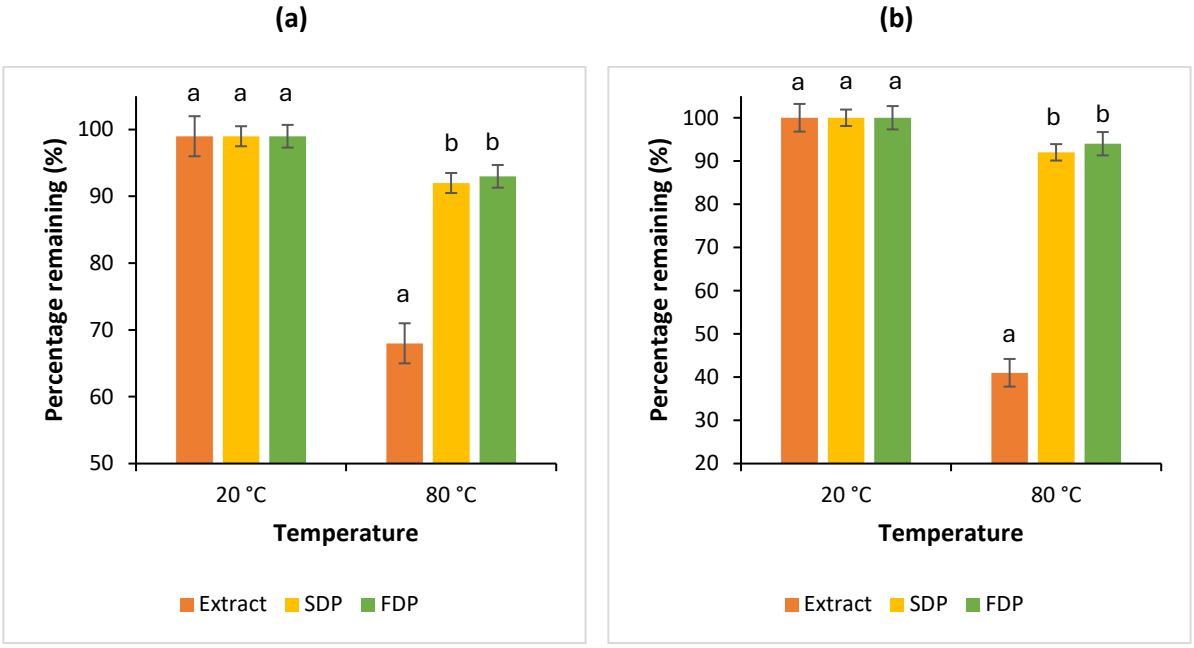

978

979 **Figure 8.** The average percentage of (a) total phenolic content and (b) total flavanoid content
980 remaining after incubation at different temperatures. Different letters on columns with the same
981 condition (temperature) suggest a statistically significant difference ($p < 0.05$).

982

983 **Tables**

984

985 **Table 1:** Extraction variables and codes of the optimization experiment.

Independent Variables	Coded variables		
	-1	0	+1
Solvent ratio (%), X1	0	40	80
Extraction temperature (°C), X2	20	40	60
Time (min), X3	15	30	45

986

987

988 **Table 2:** Spray-drying variables and codes of the optimization experiment.

Independent Variables	Coded variables				
	-1.41(-α)	-1	0	+1	+1.41(+α)
Inlet temperature (°C)	109.64	120	145	170	180.35
MD ratio (%)	39.6	50	75	100	110.3

989

990

991 **Table 3:** Coded Box-Behnken design conditions of extraction optimization and responses of total
992 phenolics, flavonoids, and anthocyanins

Run	Coded values			Analytical responses		
	X1	X2	X3	Y1	Y2	Y3
1	0	0	0	1573.14	286.00	12.82
2	-1	0	-1	1328.57	118.69	13.63
3	0	0	0	1568.57	285.82	16.63
4	+1	+1	0	1594.29	324.22	47.89
5	+1	0	-1	1525.71	308.95	24.89
6	-1	-1	0	1362.86	129.60	12.42
7	0	+1	+1	1508.57	292.80	14.63
8	0	0	0	1525.00	282.76	15.33
9	-1	+1	0	1225.71	135.71	18.64
10	0	-1	-1	1748.57	260.51	12.62
11	0	-1	+1	1551.43	256.15	10.82
12	-1	0	+1	1260.00	101.24	14.83
13	0	+1	-1	1620.00	301.00	14.43
14	0	0	0	1500.00	300.00	14.03
15	+1	-1	0	1654.29	292.36	43.08
16	+1	0	+1	1500.00	308.95	45.09
17	0	0	0	1540.00	290.20	14.73

993 X1: ethanol percentage, %; X2 temperature, ^{o}C; X3: Time, min; Y1: TPC, mg/kg, Y2: TFC, mg/kg; Y3:
994 TAC, mg/kg.
995
996

997 **Table 4:** Coded Box-Behnken design conditions of spray-drying optimization and responses of total
998 phenolics, flavonoids, and anthocyanins

Run	Coded values		Analytical responses		
	X1	X2	Y1	Y2	Y3
1	0	0	947.33	36.07	3.54
2	-1	-1	934.24	44.61	3.02
3	-1	+1	811.32	41.41	3.95
4	0	0	950.22	35.57	3.93
5	0	-1.41	973.71	55.33	2.79
6	-1.41	0	894.59	43.88	5.29
7	+1.41	0	967.59	49.86	3.45
8	0	+1.41	889.41	45.71	5.24
9	0	0	910.44	37.9	3.87
10	0	0	899.21	38.78	3.75
11	+1	-1	1005.63	48.22	4.03
12	+1	+1	852.20	47.63	2.46
13	0	0	915.22	36.21	3.85

999 X1= Temperature, °C; X2= MD percentage, %; Y1: TPC, mg/kg, Y2: TFC, mg/kg; Y3: TAC, mg/kg.

1000

1001

1002 **Table 5:** Total phenolic (TPC), flavonoid (TFC), and anthocyanin (TAC) content of microcapsules with
1003 percentage yield (PY) and encapsulation efficiency (EE).

Samples	TPC (mg/kg)	TFC (mg/kg)	TAC (mg/kg)	PY (%)	EE (%)
FDP	1042.33 [a] ±100.53	50.30 [a] ±0.51	11.77 [a] ±0.86	98.61 [b] ±0.10	92.72 [a] ±0.22
FDM	867.72 [b] ±10.58	44.95 [b] ±0.72	8.56 [b] ±1.56	98.51 [b] ±0.06	91.78 [a] ±0.84
SDP	998.35 [a] ±3.64	49.29 [a] ±0.69	4.29 [c] ±0.24	68.52 [a] ±0.04	91.20 [a] ±1.82
SDM	846.06 [b] ±0.50	41.14 [b] ±0.20	2.51 [d] ±0.12	68.47 [a] ±0.07	90.56 [a] ±0.22

1004 Data reported as mean ± standard deviation. No statistical difference exists between the means shown
1005 with the same letter in the same column ($p > 0.05$).
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1008 **Table 6:** Particle size of microcapsules.

Samples	D 3,2 (µm)	D 4,3 (µm)	D(10) (µm)	D(50) (µm)	D(90) (µm)
FDP	29.40 [a] ±0.75	80.60 [a] ±1.35	14.50 [a] ±0.20	63.57 [a] ±1.56	171.00 [a] ±2.00
FDM	15.73 [b] ±0.15	59.97 [b] ±0.78	10.20 [b] ±0.10	46.17 [b] ±0.40	131.00 [b] ±2.00
SDP	2.57 [c] ±0.02	10.60 [c] ±0.01	1.78 [c] ±0.03	8.55 [c] ±0.03	21.70 [c] ±0.20
SDM	1.76 [d] ±0.01	6.6 [d] 6±0.06	0.70 [d] ±0.01	5.38 [d] ±0.03	13.30 [d] ±0.20

1009 Data reported as mean ± standard deviation. No statistical difference exists between the means shown
1010 with the same letter in the same column ($p > 0.05$).
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1013 **Table 7:** Bioaccessibility and release of flavonoids from extract and microcapsules during different
1014 stages of *in vitro* digestion.

Samples	GD (%)	IN (%)	OUT (%)
Extract	33.42 [a] ±4.38	15.31 [a] ±3.82	8.40 [a] ±3.65
FDP	4.06 [b] ±2.04	40.13 [b] ±2.23	31.00 [b] ±3.04
FDM	2.94 [b] ±1.85	47.23 [c] ±1.20	40.45 [b] ±1.60
SDP	4.17 [b] ±1.39	47.71 [c] ±3.10	45.29 [b] ±2.36
SDM	3.00 [b] ±1.64	49.57 [c] ±2.04	22.40 [c] ±2.27

1015 Data reported as mean ± standard deviation. No statistical difference exists between the means shown
1016 with the same letter in the same column ($p > 0.05$).
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