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Effect of spray drying conditions on physicochemical and functional properties of apple cider vinegar powder

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Abstract

Background: Apple cider vinegar (ACV) contains many health benefits due to its antioxidant and acetic acid content. However, the adverse effects of directly consuming the vinegar should be eliminated to make it possible to regulate its intake as a dietary supplement. The objective of the study is to optimize the spray drying operating conditions to produce ACV powder with health benefits. A central composite design (CCD) of response surface methodology (RSM) was used to optimize the spray drying process of ACV. The influences of inlet temperature (130°C–170°C), gum Arabic concentration (5%–15% w/w), and feed flow rate (1–2 kg/h) on some product and process aspects were investigated. Spray-dried powders were evaluated for their moisture content, process yield, acetic acid content, solubility, and antioxidant content.

Results: The relationships between the model factors and final powder properties were established with ANOVA and multiple regression analysis. The model factors affected the investigated powder properties at different significance levels, where carrier concentration was the main influencing factor. Gum Arabic contributed to the retention of volatile and heat-sensitive compounds providing health benefits. All powders showed high solubility (>94%) and high antioxidant recovery, and most treatments achieved a drying yield of higher than 50%.

Conclusion: Optimization of spray drying process was achieved with application of RSM and relationships between model factors and powder properties were established. This study highlighted the feasibility of the development of ACV-based functional powder as a dietary supplement.

KEYWORDS
apple cider vinegar, functional food development, process optimization, response surface methodology, spray drying

INTRODUCTION

Apple cider vinegar (ACV) is produced by two-stage fermentation of apples, where the yeast ferments sugar to alcohol, and then Acetobacter bacteria convert alcohol into acetic acid. There have been different studies suggesting the therapeutic use of ACV due to its acetic acid content. Other ACV constituents contributing to its therapeutic effects are antioxidants, amino acids, vitamins, mineral salts, and nonvolatile organic acids.[1]

Earlier studies suggest that ACV can help in weight loss and type-2 diabetes management by lowering blood sugar and blood lipid levels.[2–4] It was also reported that acetic acid, which is the main component of ACV, slows down gastric emptying, prevents complete digestion of starch, and decreases the disaccharide activity in the small intestine.[5] Moreover, its anti-glycemic properties assist in increasing satiety and delaying gastric emptying.[4]

Despite the health benefits of ACV, its sour taste and acidity limit its consumption in fresh form. Additionally, adverse health effects (e.g., tooth erosion) can be observed in long term due to unregulated daily consumption of ACV.[6] Therefore, an ACV-based dietary supplement alternative has been introduced. The idea of the product is to deliver the ACV health benefits to consumers while avoiding the negative influences of its direct consumption. Considering the ease of handling, transportation, storage, and standardization,[7,8] microencapsulation of ACV bioactives into a soluble powder form can offer a convenient option for consumers. Alternatively, the ACV powder can be used in food formulations to improve taste and functionality, where liquid ingredients are not desired.

Spray drying is a common drying process, where liquid feed is directly converted into a powder form. It has been widely used as an encapsulation method in the food industry due to its low processing cost, operational flexibility, and applicability to heat-sensitive products.[9,10] A spray drying process consists of four stages: the atomization of feed into droplets, droplet–air contact, evaporation of water, and separation of the dried product from the drying air in a cyclone.[11] Previous studies have emphasized that encapsulation can improve delivery, protect sensitive nutrients, and mask undesired flavors.[12] In this way, the strong taste of ACV can be diminished, while the desired bioactive compounds are retained in powder, which makes the target product more desirable for dietary consumption.

The selection of wall material for spray drying is important to obtain a high-quality powder and avoid technological problems.[9,11] The presence of organic acids and low molecular weight sugars leads to powder stickiness in the drying chamber, which lowers the process yield and product quality.[13,14] The addition of wall material with a high glass transition temperature (Tg) overcomes this common spray drying problem while facilitating the encapsulation of sensitive components.[15] Gum Arabic and maltodextrin are commonly used carriers for spray drying due to their low viscosity and high solubility properties.[16] However, prior studies regarding maltodextrin digestion in the human body suggested that rapid absorption of glucose from this carbohydrate causes an elevated glycemic load.[17] In this study, gum Arabic was chosen as the wall material because of its prebiotic nature, which can support the health benefits of the powder.[18] Moreover, gum Arabic contributes to a reduction of blood glucose levels, which promotes the desirability of the end product.[19]

Spray-dried powder quality depends on many factors such as processing parameters (e.g., inlet temperature, atomizer speed, pump rate) and feed properties (e.g., viscosity, carrier type) as well as their complex interactions.[20] Therefore, statistical optimization tools like RSM have been widely implemented to understand the relationships between spray drying factors and the properties of powder.[8]

Although production and characterization of a broad range of powders from acid-rich foods have been the subject of many studies, to the best of our knowledge, no prior studies have examined the spray drying process of ACV. In this study, the main objective is to optimize the spray drying operating conditions for the production of quality ACV powder with health benefits. The focus of the end product is its acetic acid content, which is mainly responsible for the aforementioned effects on the human body. For optimization, response surface methodology was implemented for attaining the optimum process parameters for each desired response (i.e., acid acetic and bioactive content). The results of this study provide an essential basis for future investigations on the development of an ACV-based functional product.

MATERIALS AND METHODS

Materials and chemicals

Apple cider vinegar with "mother" was purchased from a local supplier. Gum Arabic was obtained from Konserval Pharmacon GmbH (Trittau, Germany). K-ACETRM acetic acid assay kit was purchased from Megazyme (Bray, Ireland). Other chemicals, that is, Folin–Ciocalteu, 2, 2-diphenyl-1-picrylhydrazil (DPPH), butylated Hydroxytoluene (BHT), sodium carbonate, and ethanol were obtained from Merck (Darmstadt, Germany).

Sample preparation

ACV was filtered through a 500-μm sieve to remove the bulky bacterial culture (mother) to avoid any clogging problems during atomization. Then, the filtered ACV was mixed with gum Arabic at different concentrations using a laboratory stirrer (EUROSTAR digital, IKA®, Staufen, Germany) to obtain 1 kg of feed solution for drying (see Section 2.4).

Spray drying

The spray drying process was performed in a pilot-scale spray dryer (Niro Atomizer Mobile Minor, GEA, Søborg, Denmark) with a concurrent flow regime. The spray dryer was equipped with a laboratory-scale peristaltic pump and a flowmeter. A rotary disk atomizer was used at a fixed speed of 23,503 g. The inlet temperature (116°C–184°C) and feed flow rate (0.7–2.3 kg/h) varied while the drying air flow rate was 80 kg/h for all experiments. Before and after every spray drying process, deionized water was run in the system for 10 min for conditioning. The dried powders were vacuum-packed and stored at 4°C inside a desiccator.

Experimental design and statistical analysis

RSM with a rotatable central composite design was used to investigate the effect of three factors on various powder characteristics. Inlet temperature (130°C–170°C), feed flow rate (1–2 kg/h), and carrier concentration (5%–15% w/w) were determined to be the independent model variables. The design had 20 randomized runs, six

TABLE 1 Experimental design for spray drying of ACV.

Pattern	Inlet temperature (°C)	Gum concentration (% w/w)	Feed flow rate (kg/h)
000	150	10	1.5
000	150	10	1.5
+−+	170	5	2
000	150	10	1.5
00a	150	10	0.7
000	150	10	1.5
−+−	130	15	1
000	150	10	1.5
−−−	130	5	1
−++	130	15	2
++−	170	15	1
−−+	130	5	2
+++	170	15	2
0a0	150	1.6	1.5
0A0	150	18.4	1.5
A00	184	10	1.5
00A	150	10	2.3
+−−	170	5	1
000	150	10	1.5
a00	116	10	1.5

central points, and each variable had three levels with two axial points (See Table 1).

The generalized polynomial model proposed for predicting the response variables as a function of the model factors is given by Equation 1

$$Y_i = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 \quad (1)$$

where Y_i is the predicted response value by the model; b_0 is a constant; b_1, b_2, and b_3 are the regression coefficients for the linear effects; b_{11}, b_{22}, and b_{33} represent the quadratic effects; and b_{12}, b_{13}, and b_{23} are for interaction effects of factors. Lastly, x_1, x_2, and x_3 are the independent variables. The analysis of variance (ANOVA), lack of fit test, determination of the regression coefficients were carried out, and the model's goodness of fit was analyzed using JMP® 14 software (SAS Institute Inc., Cary, NC). All tests were conducted in duplicates and the results were expressed by the mean value ± standard deviation (SD).

Analytical methods

Moisture content

Exactly 1 g of powder was weighed and put in a drying oven at 105°C overnight.

For the moisture content of feed solutions, the official A.O.A.C. method for vinegar was used.[21] Briefly, 10 g of feed was weighed into a 50-mm-diameter Petri dish and was evaporated on a boiling water bath for 30 min. The concentrated feed was then dried in an oven at 100°C for 2.5 h. All dried samples were cooled in a desiccator before weighing.

The moisture content (%) was calculated based on the weight difference.

Solubility

The solubility test was conducted according to the method suggested by Cano-Chauca et al.[22] with some modifications. Exactly 0.4 g of powder was dissolved in 40 mL of deionized water for 10 min using a magnetic stirrer. Afterward, the solution was centrifuged at 3000 *g* for 5 min. Twenty-five milliliter of the supernatant was collected and dried in an oven at 105°C until constant weight. Solubility was calculated based on the weight difference and the result is expressed as the percentage of solubilized materials.

Acetic acid content

Exactly 0.5 g of powder was dissolved in 50 mL deionized water using a magnetic stirrer. A commercial manual assay kit (K-ACETRM, Megazyme, Bray, Ireland) based on spectrophotometric measurement was used for the determination of acetic acid content in the powders. The absorbance values were converted into the unit of $g_{\text{acetic acid}}/100\ g_{\text{powder}}$ using the software provided by the company (MegaCalc, Megazyme, Bray, Ireland).

For the spray drying feed, the acetic acid content was determined after the required dilution, and the same assay kit was used.

A titration test was used as a reference method, where the titratable acidity of the powders was determined using 0.5 M NaOH. For that purpose, 0.5 g powder was dissolved in 50 mL of deionized water. The solution was diluted until obtaining a neutral color and then phenolphthalein was added as the color indicator. The results were calculated according to the spent volume of NaOH (0.5 M) and presented as $g_{\text{acetic acid}}/100\ g_{\text{powder}}$.

Process yield

The process yield (%) was calculated according to Equation 2:

$$Yield\% = \frac{g_{powder} \times dm_{powder}}{g_{feed} \times dm_{feed}} x\,100 \quad (2)$$

where g_{powder} and g_{feed} represent the weight of the powder and feed, dm_{powder} and dm_{feed} represent the dry matter content of powder and feed, respectively.

Antioxidant content

DPPH and Folin–Ciocalteu tests were conducted using the procedures described by Siacor et al.,[23] with some modifications. DPPH test was used to check total antioxidant capacity (TAC), while Folin–Ciocalteu test was applied for the determination of total phenolic content (TPC) retained in powders. For sample preparation, 0.5 g of powder was dissolved in 50 mL deionized water. The same procedures were applied to the feed solutions, where they were diluted 10 times before the tests.

Free radical assay—DPPH test

A 0.1 mM DPPH solution was prepared by dissolving 3.94 mg DPPH in 100 mL ethanol. A sample of 50 μL was mixed with 1.45 mL of DPPH and the mixture was incubated at room temperature (~20°C) for 30 min. Absorbance was measured using a spectrophotometer (Hitachi U1500, Hitachi High-Tech Corporation, Tokyo, Japan) at a wavelength of 517 nm. A standard calibration curve was prepared with BHT, and the results are expressed as mg BHT equivalent antioxidant capacity (BHTE) per g of powder. The radical scavenging activity was calculated using Equation 3.

$$\%DPPH\ reduction = \left(1 - \left(\frac{A_i}{A_f}\right)\right) \times 100 \tag{3}$$

where A_i is the absorbance of the sample and A_f is the absorbance of the blank (deionized water instead of the sample).

Folin–Ciocalteu test

Exactly 750 μL of 0.2 M Folin reagent was added to 100 μL of the sample solution in a 1.5 mL Eppendorf tube and mixed vigorously. After 5 min, 750 μL of 7.5% Na_2CO_3 solution was added and mixed again. The solutions were incubated at room temperature (~20°C) for 90 min and then the absorbance values were measured at a wavelength of 725 nm. Gallic acid was used for the preparation of the standard calibration curve and results were expressed as mg Gallic acid equivalent (GAE) per g powder.

Retention of bioactives

Retention of acetic acid and antioxidant contents after the spray drying process was calculated based on dry matter.[24] The general equation can be seen as Equation 4.

$$\%Retention = \frac{Bioactive_{Powder}\left[\frac{unit}{g}\right]}{Dry\,Matter_{Powder}\left[\frac{g}{g}\right]} \div \frac{Bioactive_{feed}\left[\frac{unit}{mL}\right]}{Dry\,Matter_{feed}\left[\frac{g}{mL}\right]} \times 100 \tag{4}$$

where *unit* can be mg BHTE, mg GAE, $g_{acetic\ acid}$ depending on the investigated test.

Particle morphology

Particle morphology was evaluated using Quanta FEG 250 scanning electron microscope (SEM) (FEI, Oregon, United States). A small amount of powder was fixed on SEM stubs with double-sided adhesive tape. The fixated sample was gold-coated by applying a current of 20 nÅ for 15 s (10 nm) under vacuum conditions. Afterward, the SEM images were obtained at 2 kV using 1000× and 250× magnifications.

RESULTS AND DISCUSSION

Moisture content

The moisture content of the powders varied from 6% to 11% (w/w) depending on the processing parameters. Inlet temperature, gum concentration, and feed flow rate showed a very significant ($p < 0.001$) effect on the final moisture content of the powders.

As can be seen in Figure 1a,b, increasing the gum concentration or the inlet temperature led to a decrease in moisture content, while increased feed flow rate favored higher moisture retention.

Spray drying at elevated temperatures facilitates a better heat transfer efficiency between atomized droplets and drying air in the chamber, which results in increased water evaporation rates.[22,25] These results are in line with previous spray drying studies conducted with amla juice,[26] watermelon juice,[27] and cactus pear juice cactus.[28] Moreover, increased gum Arabic concentration in the feed also showed a reducing effect on powder moisture content. It can be related to the fact that increased solid content in feed solution results in less amount of water to be evaporated during drying.[26,29]

On the other hand, using elevated feed flow rates induced higher moisture retention in the spray-dried powders. Increased moisture content in powders due to increasing feed flow rate was also observed in previous studies on acai juice.[14] It is a consequence of the reduced contact time between atomized droplets and drying air, inhibiting the rate of heat and mass transfer in the process.[20] Costa et al.[20] indicated that an increase in the feed rate results in a decreased outlet temperature, which directly promotes higher moisture retention in powders.

Solubility characteristics

According to the RSM results, the only significant factor affecting the powder solubility is the gum concentration ($p < 0.001$). Even though the model finds the gum concentration factor as statistically significant, the low value of R^2_{adj} indicates that the prediction capability of the model for solubility is rather weak (See Table 3). However, all powders showed a solubility value ranging from 94.7% to 99.8%, which can be considered highly soluble.[30] The increase in the gum concentration led to only a slight decrease in solubility (5%). Thus, the

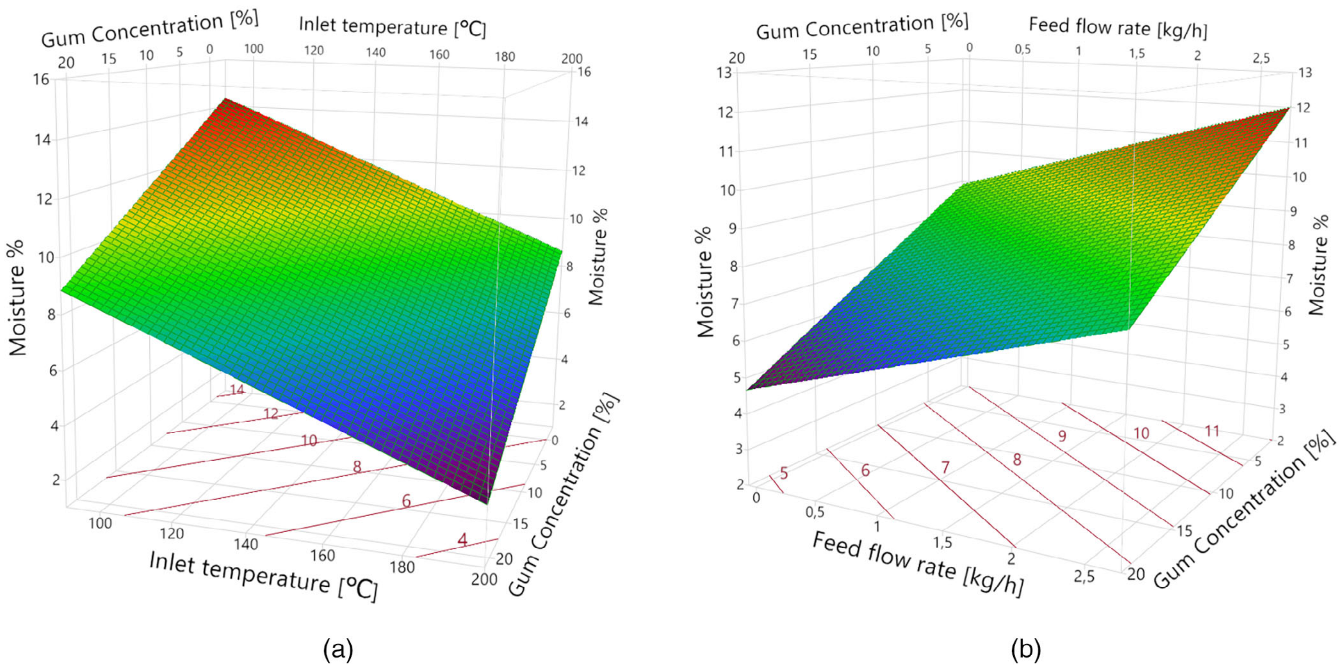

FIGURE 1 Surface plot of moisture content in the powders as a function of inlet temperature and gum concentration (a), feed flow rate, and gum concentration (b).

specific predicted value will not play a significant role in powder characteristics.

This appears to be a case of solubility difference between the gum and vinegar compounds. When the compounds with higher solubility compose the majority of powders, the solubility tends to be better.[31] Here, both vinegar and gum Arabic are highly soluble, yet vinegar has better solubility in water compared with the gum carrier. This situation leads to a slight decrease in solubility in powders when the gum concentration is increased.

Previous studies have reported that the powder solubility is mainly affected by the carrier type rather than the concentration,[32,33] which explains why using the same carrier type results in only a slight difference.

Acetic acid content

According to the RSM results, the significant model coefficients affecting the acetic acid content in powders are found to be the gum concentration and inlet temperature as well as quadratic effects of all three design parameters ($p < 0.001$). From Figure 2a,b, it is seen that there is an optimal value for acetic acid encapsulation since all factors showed a curvature effect.

The increased carrier concentration contributed to acetic acid retention in powders. In the literature, retention of a higher amount of volatiles due to increased carrier concentration was also observed in spray drying of citral and linalyl acetate.[34] Crust formation is an important factor for volatile retention during a spray drying process. It was explained that a higher carrier concentration decreases the duration of crust formation around the atomized droplets. This crust layer reduces the loss of volatiles by forming a protective semipermeable membrane. However, if the gum concentration of the feed solution is too high, volatile retention may diminish as a result of induced cracks on the protective layer.[35]

Besides the gum concentration, the inlet temperature also influences the formation of the crust layer. It was reported that increased inlet temperature promotes faster crust formation—especially in the case of high solids concentration—while high temperatures can lead to cracking on the surface.[34] Reineccius[36] also mentioned that spray drying at very high temperatures leads to "ballooning" or heat damage on the droplet. The ballooning occurs because of steam formation inside the droplet at high temperatures, which results in a thin-walled hollow particle due to expansion in droplet. In that case, the particle cannot retain the volatile compounds as good as nonballooned hollow particles.[36]

The results showed that the feed flow rate had a curvature effect on the acetic acid content of the powders. Too high feed flow rates might lead to poor crust formation due to insufficient heat and mass transfer after atomization.[20] On the other hand, one would expect an increased volatile loss because of elongated high-temperature exposure when the feed flow rate is not high enough.

Therefore, it is important to find an optimal combination of inlet temperature, gum concentration, and feed flow rate to maximize volatile retention in the spray-dried powders. According to the RSM model, the maximum acetic acid in powders can be obtained at a combination effect of inlet temperature at 140°C, 15% (w/w) gum concentration, and feed flow rate of 1.5 kg/h.

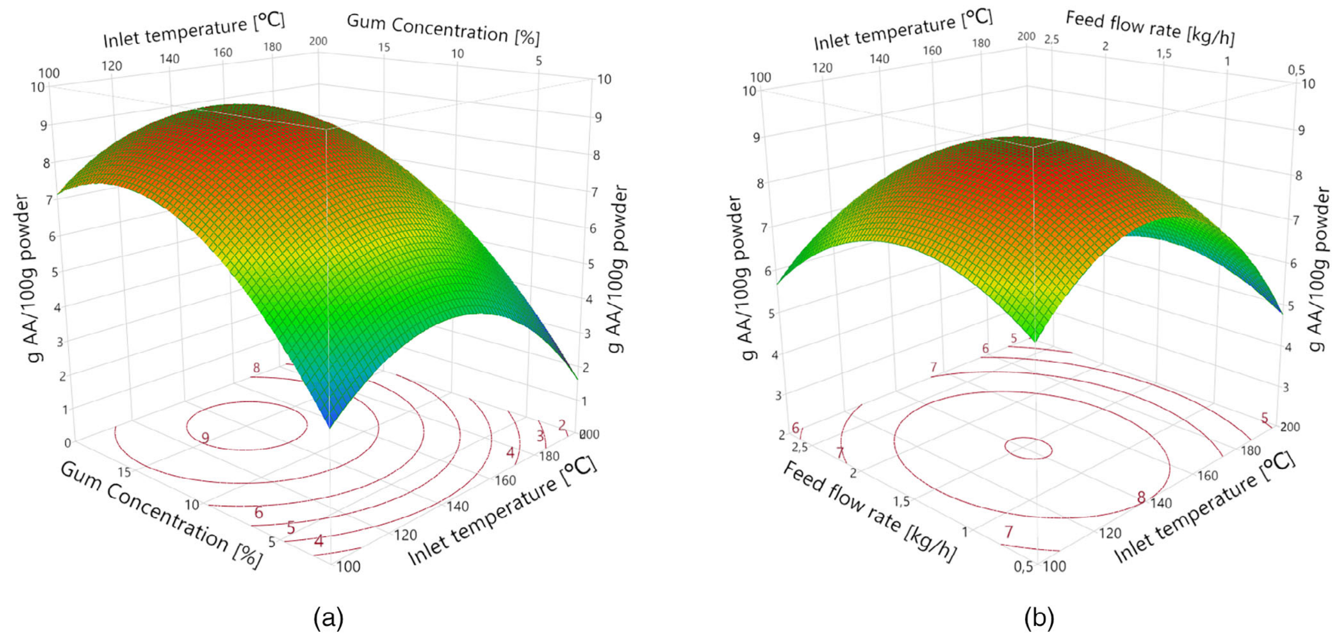

FIGURE 2 Surface plot of acetic acid content in powders as a function of (a) inlet temperature and gum concentration, (b) feed flow rate, and inlet temperature.

It should be noted that the titratable acidity and acetic acid content in powders are highly correlated according to Pearson correlation coefficient ($r = 0.96$). That means acidity in powders is mainly from acetic acid content and the presence of other organic acids (e.g., malic acid) is negligible.

Process yield

Inlet temperature, gum concentration, and its quadratic factor showed a significant influence over the process yield. The most noticeable impact was seen with the change in gum concentration. According to Figure 3, an increase in the inlet temperature and carrier concentration leads to a higher process yield.

Similar results were observed in previous studies, where increased inlet temperature and carrier concentration led to a higher yield.[37,38] Tonon et al.[14] explained that increased inlet temperature during spray drying positively affects the process yield via increased mass and energy transfer rates between the droplets and drying air. In addition to that, an increase in the carrier concentration forms a better protective layer around the droplets and avoids sticking to the drying chamber.[11,39] According to Fazaeli et al.,[37] the positive relationship between carrier concentration and process yield stems from increased Tg values. Therefore, the increased addition of gum Arabic in the feed solution favored the obtained amount of spray-dried powder. These results are not in line with earlier findings of Tonon et al. and Ribeiro et al., who argued that increased carrier concentration causes a reduced yield due to increased feed viscosity.[14,40] It should be noted that the negative correlation between increased carrier concentration and process yield was observed when a high carrier concentration was used in the studies (>15% w/w). That explains the difference in the findings. Therefore, process yield can be improved by increasing gum concentration only up to a critical feed viscosity.

Overall, all processing conditions except 0a0 provided a successful spray drying, which was described in the literature as a process yield of over 50%.[9]

Antioxidant content

For both antioxidant analyses, the only model factor having a significant impact on the antioxidant content of powders was gum concentration and its quadratic effect ($p < 0.001$).

The RSM results showed that the gum concentration and the antioxidant content have a negative correlation. The values of radical scavenging activity ranged from 6.6 to 47.6 mg BHTE/g, while the values of TPC were between 3.5 and 15.7 mg GAE/g (see Table 2). A similar trend was obtained in spray drying studies conducted with green tea extract,[41] amla juice,[26] and cinnamon.[42] The reduction of antioxidant content with increased carrier concentration is simply a result of the dilution effect of gum Arabic addition on apple cider vinegar.[41]

As illustrated in Table 2, the retention of polyphenols and total antioxidants was above 100% for all treatment conditions except for 0a0. This exception is related to insufficient encapsulation originating from too low carrier concentration (1.6% w/w). The increase in antioxidants after spray drying was also observed previously observed in studies on cranberry juice and cactus pear juice.[24,43] According to Zhang et al.,[24] the high retention of antioxidants in cranberry juice was associated with its relatively thermal stable polyphenols, which indicates the importance of thermal stability of antioxidants for better recovery in a spray drying process. Increased antioxidant amount after the process was also attributed to the conformational changes in antioxidant structure throughout the spray drying process. It was suggested that these chemical changes improve solubilization and extraction of the antioxidants, which increases their detectability in the analyses.[24]

Even though increased carrier in the feed decreased the antioxidant content of the powders, the recovery values from TPC and TAC did not show a significant difference ($p > 0.05$). This result supports that the encapsulation of ACV antioxidants by gum Arabic is effective at the investigated concentrations.

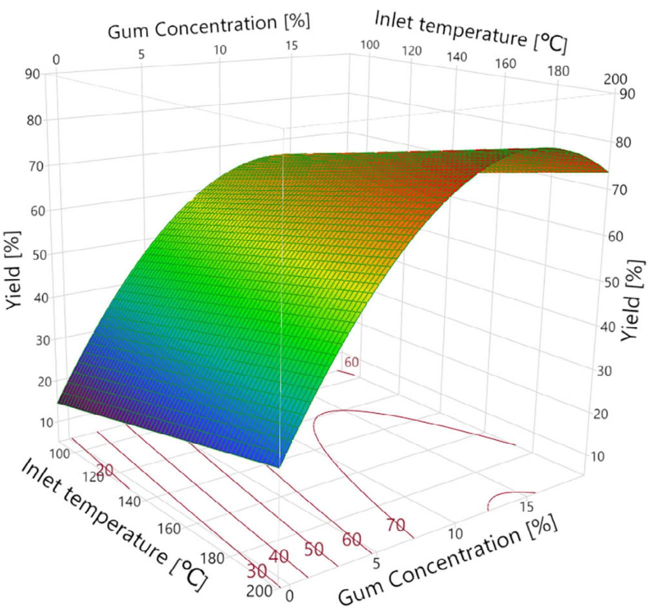

FIGURE 3 Surface plot of process yield as a function of inlet temperature and gum concentration.

Response surface analysis

For model simplification, only the factors having a significance level of higher than 5% were included and the resulting simplified equations were evaluated for sufficiency and fitness by ANOVA. Table 3 illustrates the regression coefficients, determination coefficients (R^2_{adj}), and p-values of the models for the simplified models.

The fitted models indicated no lack of fit, showed significant regression and satisfactory determination coefficients—except for the solubility. That means—excluding the model for solubility—more than 79% of the response variable can be explained as a function of the three studied factors. The high value of the determination coefficient

TABLE 2 Values of solubility, AOC, TPC, and retention of antioxidants in different treatments.

Pattern	Solubility [%]	mg BHTE/g_{powder}	mg GAE/g_{powder}	AOC RT%[a]	TPC RT%[a]
000	97.2	17.7 ± 1.5	6.2 ± 0.1	173.4 ± 14.7	120.6 ± 6.2
000	95.3	16.1 ± 1.6	6.1 ± 0.6	156.3 ± 15.4	116.1 ± 6.0
+−+	99.0	27.5 ± 0.3	8.6 ± 0.3	148.3 ± 1.6	104.6 ± 1.4
000	97.8	16.1 ± 1.4	5.6 ± 0.0	160.2 ± 14.0	110.2 ± 5.7
00a	98.2	14.0 ± 0.1	5.9 ± 0.1	135.6 ± 1.3	113.5 ± 5.9
000	98.9	15.4 ± 0.5	5.3 ± 0.1	152.3 ± 5.1	103.7 ± 5.4
−+−	96.2	9.4 ± 0.7	4.2 ± 0.5	137.4 ± 10.1	117.0 ± 3.4
000	98.4	15.2 ± 0.5	7.1 ± 1.3	150.4 ± 5.2	138.8 ± 6.1
−−−	98.9	24.4 ± 0.9	9.4 ± 0.5	133.2 ± 5.3	114.9 ± 1.5
−++	96.6	9.7 ± 0.5	4.7 ± 0.7	143.8 ± 8.2	130.5 ± 3.8
++−	96.3	9.9 ± 0.1	4.1 ± 0.1	142.8 ± 1.6	112.2 ± 3.3
−−+	99.8	23.7 ± 0.4	8.8 ± 0.1	130.5 ± 2.1	109.2 ± 1.5
+++	95.6	8.3 ± 0.3	4.5 ± 0.5	120.6 ± 4.8	123.2 ± 3.6
0a0	99.8	47.6 ± 1.5	15.7 ± 0.3	122.8 ± 3.8	93.8 ± 1.8
0A0	94.7	6.6 ± 0.3	3.5 ± 0.0	129.4 ± 5.2	119.5 ± 4.4
A00	96.0	16.0 ± 0.5	6.3 ± 0.1	152.9 ± 5.0	118.9 ± 6.2
00A	94.9	12.7 ± 0.5	5.9 ± 0.3	127.1 ± 4.6	116.9 ± 6.1
+−−	97.1	26.0 ± 0.7	9.6 ± 0.2	138.2 ± 3.9	115.6 ± 1.5
000	95.1	15.5 ± 0.6	6.0 ± 0.3	152.8 ± 6.2	117.2 ± 6.1
a00	95.9	12.7 ± 0.1	5.2 ± 0.1	127.6 ± 0.8	103.3 ± 5.3

[a]AOC RT%: Retention percentage of antioxidant content in powders, TPC RT%: Retention percentage of total polyphenol in powders.

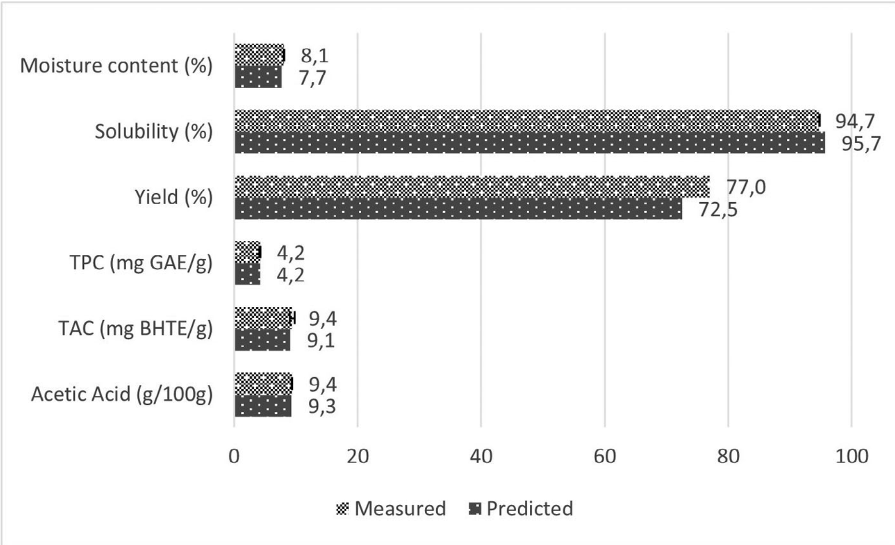

FIGURE 4 Comparison of measured and predicted values of the responses at optimum conditions for spray-dried ACV powder.

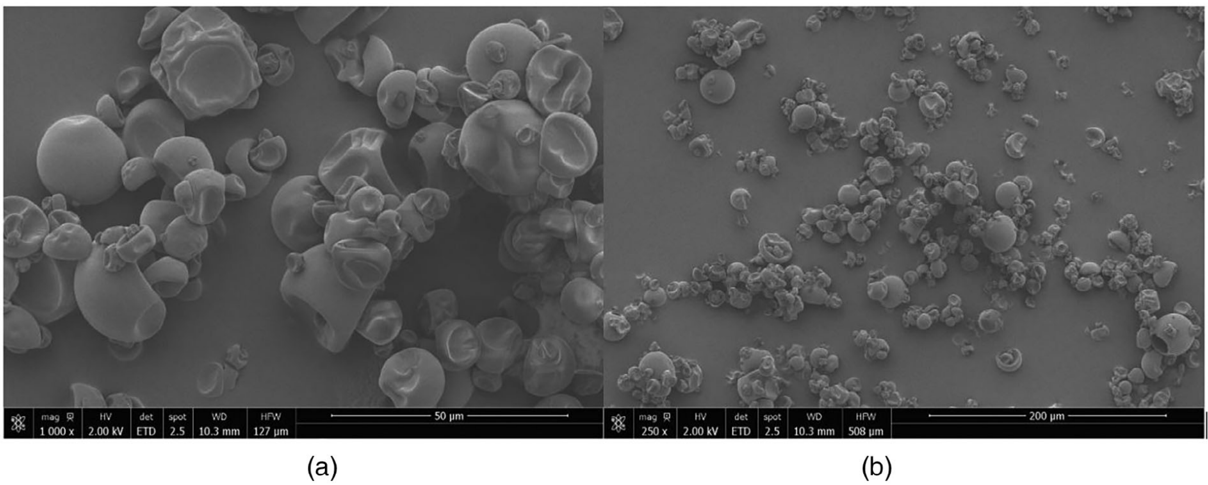

FIGURE 5 SEM images of spray-dried ACV powder at optimized conditions for acetic acid retention. 1000× (a) and 250× (b) magnification.

indicates the suitability of the models to be used for prediction purposes. The sufficiency of the model equations was checked by comparing the predicted and experimental values (see Figure 4) and concluded to be satisfactory including the solubility model. As it was mentioned earlier, all samples showed a solubility value of higher than 94%, which implies that solubility characteristics do not provide a considerable differentiation among the powders. Therefore, the RSM model still provides a reliable prediction even including the solubility factor.

To conclude, the obtained RSM model is acceptable for the optimization of desired bioactive retention in ACV powder as well as suitable for prediction. The optimized conditions for the purpose of the end product are determined to be inlet temperature of 140°C, gum Arabic concentration of 15% (w/w), and feed flow rate of 1.5 kg/h.

Powder morphology

Figure 5 shows the SEM images of ACV powder produced at optimized conditions designed for maximum acetic acid retention. Depending on various factors such as inlet temperature and carrier material, particle size and shape may alter during spray drying.[11] In this study, particles showed irregularly spherical structures of various sizes, which is typically seen in spray-dried materials. Daza et al.[11] also reported similar results when they used gum Arabic as wall material in spray drying of Cagaita fruit extracts.

Particles showed a hollow-shaped structure due to quick shell formation, which is a common characteristic for skin-forming materials.[9,44] A hollow-shaped structure is useful for bioactive encapsulation, and it was also reported in the literature when gum Arabic was used as the carrier.[9]

TABLE 3 Regression coefficients and adjusted R^2 for the final reduced models.

Regression coefficient	Moisture content (%)	Solubility (%)	Acetic acid content (g/100 g)	Process yield (%)	TPC (mg GAE/g)	TAC (mg BHTE/g)
Constant						
b_o	8.3245	97.094	8.5637	70.3675	6.0302	15.4617
Linear						
b_1	−1.0470	ns	−0.3457	2.6283	ns	ns
b_2	−1.1467	−1.3723	1.3085	9.5807	−2.4085	−8.1104
b_3	0.5553	ns	−0.0438	ns	ns	ns
Square						
b_1^2	ns	ns	−0.3671	ns	ns	ns
b_2^2	ns	ns	−0.6483	−6.0870	0.6209	1.7701
b_3^2	ns	ns	−0.2787	ns	ns	ns
Interactions						
b_{12}	ns	ns	ns	ns	ns	ns
b_{13}	ns	ns	ns	ns	ns	ns
b_{23}	ns	ns	ns	ns	ns	ns
R^2_{adj}	0.81	0.41	0.91	0.79	0.91	0.91
p (model)	<0.0001	0.0006	<0.0001	<0.0001	<0.0001	<0.0001

Note: b_i: the estimated regression coefficient for the main linear effects. b_i^2: the estimated regression coefficient for the quadratic effects. b_{ij}: the estimated regression coefficient for the interaction effects. $i = 1$: inlet temperature; $i = 2$: gum Arabic concentration; $i = 3$: feed flow rate.
Abbreviation: ns, nonsignificant.

The stability of the retained bioactives is directly influenced by particle morphology.[24,45] Cracks or fissures on the surface may diminish the protection of the core material due to increased gas permeation.[24] According to Figure 5, the particle surfaces are mostly intact besides a few broken particles, which is an indication of good protection of the core material.

Furthermore, Zhang et al.[9] mentioned that the surface roughness of the particles decreases wall deposition due to decreased adhesive forces between two contacting surfaces, which could be one of the reasons for the high process yields obtained with increased gum concentration in this study.

CONCLUSION

This study investigated the influence of wall material concentration, inlet temperature, and feed flow rate on physicochemical properties of ACV-based powder using a central composite design. The fitted equations provided good insight regarding the effects of inlet temperature, gum concentration, and feed flow rate on spray-dried ACV powder properties. The model obtained using RSM showed a good prediction capability regarding process yield, acetic acid, moisture, and antioxidant content.

In the framework of the study, the targeted ACV-based functional product could be developed with the desired bioactive content by optimizing spray drying conditions using gum Arabic as the carrier. However, further research is needed for improving the taste and powder handling properties of spray-dried powders to provide a user-friendly end product. This study offers theoretical knowledge for the scale-up and industrialization of ACV-based functional powders.

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CONFLICT OF INTEREST STATEMENT

The authors declared no conflict of interest.

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