Research Article

Optimization of *Agave tequilana* Weber var. *Azul* Juice Spray Drying Process

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Received 21 August 2013; Revised 15 November 2013; Accepted 15 November 2013; Published 29 January 2014

Academic Editor: Souhail Besbes

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In this work, the response surface methodology was employed to optimize the microencapsulation of *Agave tequilana* Weber var. *azul* juice with whey protein isolated using a spray drying technique. A Box-Behnken design was used to establish optimum spray drying conditions for *Agave tequilana* juice. The process was optimized to obtain maximum powder yield with the best solubility time, hygroscopicity, bulk density, water activity, and reducing sugars. The independent parameters for the spray drying process were outlet temperature of 70–80°C, atomizer speed of 20000–30000 rpm, and airflow of 0.20–0.23 m³ s⁻¹. The best spray drying condition was at outlet temperature of 80°C, atomizer speed of 20000 rpm, and air flow rate of 0.23 m³ s⁻¹ to obtain maximum powder yield (14.65%_{bm}), minimum solubility time (352.8 s), maximum bulk density (560 kg m⁻³), minimum hygroscopicity (1.9 × 10⁻⁷ kg_{water} s⁻¹), and minimum *a_w* (0.39). The *Agave tequilana* powder may be considered as an interesting source of dietary fiber used as food additive in food and nutraceutical industries.

1. Introduction

Agave tequilana Weber var. *azul* is an important crop in the state of Jalisco, México, for tequila production. Today, *Agave* juice is also used to make syrup and fructooligosaccharides (FOS) powder as another alternative industrial due to its high content of fructans. These *Agave* fructans consist of a complex mixture of FOS containing principally $\beta(2-1)$ and $\beta(2-6)$ linkages [1, 2], which can stimulate the growth of bifidobacteria as prebiotic, increase Ca⁺⁺ absorption, and decrease blood triglyceride levels [3–6]. The prebiotic effect opens the new alternatives for *Agave* fructans as food ingredients: sweeteners, texture modifiers, and fat-replacer in food products [7]. Also, FOS are officially recognized as natural food ingredients and are classified as dietary fibers. However,

the FOS are carbohydrates that undergo many changes, like the hydrolysis, Maillard-reaction, and caramelization [3].

Spray drying is one of the most important methods for obtaining powders. Although the spray drying is a fast process, changes on the spray drying conditions can affect the physicochemical and functional proprieties. For example, the spray drying of chicory inulin at a temperature range of 135–195°C resulted in significant FOS degradation (20 to 100%) and loss of its functional properties because the heat induced degradation [8, 9]. Microencapsulation of FOS in a carrier is an alternative technique that can be used to minimize degradation and loss of functional properties during spray drying [10, 11]. Spray drying is the most commonly used encapsulation method in the food industry [11] and exist carriers for several spray-dried products [12, 13]. The influence

of the main process variables, such as temperature, atomizer speed, air flow, feed flow, nature of food and its geometry, carriers types, and solution to sample ratio on the mass transfer mechanism, has been studied extensively [14–18].

Response Surface Methodology (RSM) will be a useful tool to obtain successful spray drying operating parameter, because RSM describes the effect of the test variables on the responses, determines interrelationships among test variables, and represents the combined effect of all test variables on the response [19, 20]. Moreover we studied out the effects of feed properties and drying conditions on the physical properties of the powder, like moisture content, bulk density, hygroscopicity, solubility time, and flow behavior. We found that inlet air temperature and feed flow rate are the significant parameters in case of most of the responses analyzed by [19]. It was found that the air temperature and airflow rate are the important parameters in case of most of the responses on ginger extract spray drying [21]. We found that the best drying conditions for the inulin were: 210°C and 5% and powder particles presented spherical and smooth surfaces [22].

Although the best drying conditions for inulin or FOS were reported by [22], additional work is needed to determine the optimal processing parameters to spray dry *Agave* juice. The purpose of this work was to optimize the spray drying process on physicochemical properties of *Agave tequilana* Weber var. *azul* fructooligosaccharides.

2. Materials and Methods

Seven-year-old *Agave tequilana* Weber var. *azul* “pine” and “head” were harvested and processed (washed, squeezed, and filtrated by Agaveros Industriales of Jalisco in Jocotepec, Jalisco, Mexico) to obtain the juice, which was kept at –20°C.

2.1. Spray Drying. A pilot scale spray dryer (GEA NIRO model A/S Production minor, Columbia, Washington, D.C., USA) with a cylindrical section of the drying chamber that is 1.2 m in diameter and 1 m high and the conical section that is 0.7 m high with a bottom outlet that is 0.3 m in diameter. The rotating disc atomizer has twenty-four annular 4×3 mm orifices on an 18 mm thick disc with a diameter of 0.10 m. The atomizer has the capacity to evaporate 40 kg of water per hour and was used for the drying process in all experimental treatment. The outlet temperature, atomizer speed, and airflow were set to 70 and 80°C, 20000 and 30000 rpm, and 0.20 and 0.23 m³ s^{–1}.

In each treatment, the *Agave* juice was defreezed at 4°C, concentrated at 20° Brix, and mixed with 0.1% commercial whey protein isolated WPC-80 (donated by America Alimentos Company). The juice mixture was homogenized using a mixer (Glas-Col Mod. Precision Stirrer, IN, USA).

The juice-whey protein isolated mixture was placed into a stainless steel container. A plastic flexible hose was placed inside the container and connected to the inlet of a variable flow peristaltic pump (Watson Marlon, Model 504U, Falmouth, Cornwall, UK). The pump outlet was then connected

to the feed hose of the atomizer. The drying time in all treatments was 45 min.

The spray-dried powders were collected, weighed, and packed in 4 L glass flasks.

2.2. Bulk Density and Solubility Time. The total of 20 ± 0.1 g of powder was transferred into a 100 mL graduated cylinder and gently dropped onto a rubber mat from a height of 0.15 m for 40 times. The bulk density was calculated by dividing the powder weight by the volume it occupied in the cylinder; samples were run in duplicate [23–26].

The spray-dried powder solubility time was determined as described by [26]. Briefly, 10 ± 0.1 g of powder was added to 250 mL distilled water at 25°C. The mixture was then agitated on a stirring hot plate (Cimarec, Model SP131015, Thermo Scientific, San Jose, CA, United States) set at position 350 rpm and the time required for the material to completely dissolve was recorded. All samples were run in duplicate.

2.3. Hygroscopicity. The total of 1 ± 0.01 g of powder was evenly spread on a glass dish (0.09 m diameter) with a high humid air-to-powder surface area ratio. Samples were then placed in desiccators set at 25°C and 85% relative humidity using an HNO₃ solution. A 90 min sampling interval was selected to obtain the moisture sorption kinetics. The weight gain of the samples was considerably lower after 90 min [23]. Thus, the weight increase per gram of powder solids after 90 min was determined [23, 26]. All samples were run in duplicate.

2.4. Water Activity. The water activity of *Agave* juice powders was measured at 25°C using an Aqualab 3TE (Decagon, Pullman, WA, USA) calibrated with a LiCl solution with known water activity. All samples were run in duplicate.

2.5. Yield. The *Agave* juice yield of all experiments was determined. The mass of product was divided by the product of the total mass of solution dried during each experiment (8.224 kg) and the total solids concentration. The average and range of yields for a particular set of operating conditions were then calculated from the three repeat experiments. To estimate the yield and for further spray-drying analysis, only the powder collected from the sample pot was considered.

2.6. Total Reducing Sugars. Direct reducing sugars were measured by the Fehling method modified [27], without hydrolysis. All samples were run in duplicate.

2.7. Experimental Design and Optimization. The effects of the three independent processing parameters, outlet temperature (X1, °C), atomizer speed (X2, rpm), and airflow (X3, m³ s^{–1}) on the dependent variables were investigated; the response surface methodology is presented in Table 1. Box-Behnken designs are response surface designs requiring only three levels, which are coded as –1, 0, and +1. A total of 17 experiments in this study were based on three levels and the three-factor experimental design, with three replicates at the centre of

TABLE 1: Box-Behnken experimental design.

Std. order	Run order	Independent variables		
		Temperature (°C)	Atomizer speed (rpm)	Air flow (ms ⁻¹)
2	1	80 (+1)	20,000 (-1)	23 (0)
14	2	75 (0)	25,000 (0)	23 (0)
8	3	80 (+1)	25,000 (0)	24 (+1)
1	4	70 (-1)	20,000 (-1)	23 (0)
15	5	75 (0)	25,000 (0)	23 (0)
9	6	75 (0)	20,000 (-1)	22 (-1)
4	7	80 (+1)	30,000 (+1)	23 (0)
7	8	70 (-1)	25,000 (0)	24 (+1)
13	9	75 (0)	25,000 (0)	23 (0)
10	10	75 (0)	30,000 (+1)	22 (-1)
11	11	75 (0)	20,000 (-1)	24 (+1)
3	12	70 (-1)	30,000 (+1)	23 (0)
6	13	80 (+1)	25,000 (0)	22 (-1)
12	14	75 (0)	30,000 (+1)	24 (+1)
5	15	70 (-1)	25,000 (0)	22 (-1)

Values in parentheses () indicate coded levels.

the design to estimate the pure error sum of squares. The statistical software MINITAB (Release 14.1) was used for the experimental design, data analysis, and regression modeling. The independent variables were X1 (70–80°C), X2 (20,000–30,000 rpm), and X3 (0.20–0.23 m³ s⁻¹) with point center at 75°C, 25000 rpm, and 0.215 m³ s⁻¹, respectively. The experimental order was randomized. Experimental data from the Box-Behnken design was analyzed and fitted to a second-order polynomial model. Consider

$$Y_k = \beta_0 + \sum_{i=1}^2 \beta_i X_i + \sum_{i=1}^2 \beta_{ii} X_i^2 + \sum_i \sum_{j=i+1}^2 \beta_{ij} X_i X_j, \quad (1)$$

where Y_k is the predicted response, β_0 is the constant (intercept), β_i is the linear coefficient, β_{ii} is the quadratic coefficient, and β_{ij} is the cross-product coefficient. X_i and X_j are independent variables.

3. Results and Discussion

3.1. Fitted Models and Response Surfaces. Results of the experimental design with standard deviation to quality parameters of *Agave* juice subjected to different spray drying conditions are shown in Table 2. The regression coefficients (β) of the quadratic polynomial equation for the coded independent variables, interactions upon response variables, determination coefficient, and lack-of-fit test and probability are shown in Table 3 for all the responses. The significant terms ($P \leq 0.05$) were used as a tool to check the significance of each of the coefficients of the proposed models for each response.

Analysis of variance showed that the quadratic polynomial models were highly significant ($P \leq 0.001$) for solubility (Y5) and yield (Y6) and less significant for bulk density (Y2), activity water (Y1), hygroscopicity (Y3), and reduced sugar (Y4). The coefficients of determination (R^2) values for the

response variables Y6 and Y5 were greater at 0.99 and 0.98, respectively; for variable Y2, the coefficient was 0.82; and for variables Y4, Y3, and Y1, the coefficients ranged from 0.66 to 0.59. The coefficients of variation (CV) should not be greater than 10%, but in this work, they were found in the range of 0.026–4.72% for all the responses, which indicates better precision and reliability of the experiments carried out. The lack-of-fit, which measures the fitness of the models, resulted in a significant F -value only for solubility and yield, indicating that these models were sufficiently accurate for predicting those responses. The P values of the models were 0.0001 for yield, 0.001 for solubility, 0.16 for bulk density, 0.558 for hygroscopicity, 0.484 for reduced sugar, and 0.638 for water activity, which further indicates the goodness of fit.

3.2. Response Surface Analysis of Powder Yield. Using multiple regression techniques, a response surface model was developed for powder yield as a function of the spray drying process variables. A complete-second order model (1) was tested for its ability to describe the response surface. The analysis of variance (Table 3) shows that the model is highly significant ($P < 0.0001$). Values of P less than 0.05 indicate that the model terms are significant. In this case, β_2 , β_3 , β_1^2 , β_2^2 , β_3^2 , β_{12} , β_{13} , and β_{23} are significant model terms. Values greater than 0.10 indicate that the model terms are not significant. The lack-of-fit P value of 0.029 implies that the lack-of-fit is highly significant. Thus, all of the quadratic terms are significant.

The powder yield varied in the range from 24.85% to 76.70%, in our experiment runs. The quadratic polynomial model was used to fit the quadratic model for this response with $R^2 = 0.990$. The ANOVA analysis for the response “powder yield” showed that atomizer speed and airflow are more significant than outlet temperature. However, in two representative plots are shown the effects of outlet

TABLE 2: Responses to the quality parameters investigated.

Std. order	Run order	Dependent variables									
		Activity water		Bulk density		Hygroscopicity $\times 10^{-7}$		Reduced sugars		Solubility	
		a_w	σ	(kg m ⁻³)	σ	(kg _{wat} s ⁻¹)	σ	(kg L ⁻¹)	σ	(s)	σ
2	1	0.352	0.013	0.556	0.000	2.011	0.038	0.005	0.004	73	0.000
14	2	0.418	0.047	0.556	0.095	2.139	0.011	0.008	0.003	41	0.001
8	3	0.484	0.067	0.556	0.029	2.000	0.017	0.004	0.011	36	0.001
1	4	0.353	0.019	0.588	0.000	2.006	0.010	0.004	0.007	58	0.001
15	5	0.415	0.010	0.500	0.051	2.153	0.015	0.009	0.003	46	0.001
9	6	0.415	0.023	0.588	0.023	1.967	0.053	0.009	0.003	53	0.001
4	7	0.343	0.006	0.556	0.009	1.944	0.015	0.017	0.006	66	0.001
7	8	0.323	0.022	0.667	0.067	2.047	0.021	0.016	0.007	55	0.001
13	9	0.358	0.010	0.521	0.007	2.014	0.053	0.005	0.010	50	0.000
10	10	0.339	0.009	0.541	0.000	1.928	0.040	0.015	0.004	65	0.001
11	11	0.418	0.054	0.625	0.000	2.050	0.016	0.016	0.004	53	0.001
3	12	0.361	0.016	0.541	0.000	1.967	0.044	0.013	0.009	69	0.000
6	13	0.344	0.008	0.541	0.000	2.025	0.041	0.005	0.010	76	0.002
12	14	0.323	0.099	0.556	0.023	2.158	0.020	0.008	0.003	38	0.002
5	15	0.342	0.005	0.541	0.007	2.008	0.023	0.003	0.004	44	0.001

σ : standard deviation.

TABLE 3: Analysis of variance of the regression coefficients of the fitted quadratic equation for the quality parameters studied.

Coefficients	Response				
	Aw (Y1)	Bulk density (Y2) Kg L ⁻¹	Hygroscopicity (Y3) Kg _{water} s ⁻¹	Reduced sugar (Y4) Kg L ⁻¹	Solubility (Y5) s
β_0 (intercept)	0.397***	0.5257***	0.7567***	7.4*	7.59***
Linear					
β_1	0.018 ns	-0.016 ns	-0.0021 ns	-0.8 ns	0.5113*
β_2	-0.0215 ns	-0.0204 ns	-0.0016 ns	2.4625 ns	0.0325 ns
β_3	0.0135 ns	0.0241 ns	0.0148 ns	1.3875 ns	-1.1638**
Cross-product					
β_{12}	-0.0043 ns	0.0117 ns	-0.0025 ns	0.7 ns	-0.755*
β_{13}	0.0398 ns	-0.0278 ns	-0.0058 ns	-3.2 ns	-2.1275***
β_{23}	-0.0047 ns	-0.0055 ns	0.0133 ns	-3.375 ns	-1.13**
Quadratic					
β_{11}	-0.0226 ns	0.0167 ns	-0.0226 ns	-1.3875 ns	1.8113**
β_{22}	-0.0221 ns	0.0179 ns	-0.0206 ns	3.7375 ns	1.7037**
β_{33}	-0.0011 ns	0.0339 ns	-0.0068 ns	0.8375 ns	-0.6087 ns
R^2	0.59	0.82	0.63	0.66	0.98
CV	0.049	0.029	0.026	4.727	0.51
Lack-of-fit	0.266	0.504	0.577	0.118	0.951
P	<0.638	<0.160	<0.558	<0.484	<0.001

* Significant at $P \leq 0.05$, ** significant at $P \leq 0.01$, and *** significant at $P \leq 0.001$.
ns: not significant.

FIGURE 1: Surface plot showing (a) the effect of temperature and atomizer speed at air flow $0.215 \text{ m}^3 \text{s}^{-1}$ and (b) the effect of temperature and air flow at an atomizer speed of 25000 rpm on the powder yield.

temperature with atomizer speed and airflow (Figures 1(a) and 1(b)). Figure 1(a) shows the effect of temperature and atomizer speed on powder yield at the airflow center point (0) and Figure 1(b) shows the effect of outlet temperature and airflow. In Figure 1(a) it is evident that at lower outlet temperatures and higher atomizer speeds the powder yield presents the higher value. Evenly in Figure 1(b) we can also observe that at lower outlet temperature and higher airflow the powder yield presents the higher value. Even if the inlet temperature was constant in all experiments (180°C), the

increase of feed flow due to control of the outlet temperature and the nature amorphous of spray-dried material causes significant problems with the deposition of powder on the wall of spray dryer, which reduces the powder yield [19, 22].

3.3. Response Surface Analysis of Solubility Time. Using multiple regression techniques, a response surface model for the powder solubility time, as a function of spray drying process variables, was developed. A complete second-order model

FIGURE 2: Surface plot showing (a) the effect of temperature and atomizer speed at an air flow of $0.215 \text{ m}^3 \text{ s}^{-1}$ and (b) the effect of temperature and air flow at an atomizer speed of 25000 rpm on the powder solubility time.

(1) was tested for its ability to describe the response surface. Analysis of variance (Table 2) shows that the model is highly significant ($P < 0.001$). Values of P less than 0.0500 indicate that the model terms are significant. In this case, β_0 , β_1 , β_3 , β_1^2 , β_2^2 , β_{12} , β_{13} , and β_{23} are significant model terms. Values greater than 0.1000 indicate that the model terms are not significant. The lack-of-fit P value of 0.951 implies that

the lack-of-fit is slightly significant. However, the determination coefficient was at 0.98.

Figures 2(a) and 2(b) present the surface and contour plots for solubility. Figure 2(a) shows the effect of temperature and atomizer speed at the airflow centre point ($0.200 \text{ m}^3 \text{ s}^{-1}$) on powder solubility. It is evident from Figure 2(a) that the solubility showed an increase with an increase in outlet

FIGURE 3: Surface plot showing (a) the effect of temperature and atomizer speed at an air flow of $0.215 \text{ m}^3 \text{s}^{-1}$ and (b) the effect of temperature and air flow at an atomizer speed of 25000 rpm on the bulk density.

temperature and atomizer speed. However, at the centre point, the solubility decreases to a lower value at 38 s. Figure 2(b) shows that with an increase in outlet temperatures the solubility is also increased, but an increase in airflows represents a decrease in the solubility. This trend is similar to that reported by other works [24, 26].

3.4. Response Surface Analysis of Bulk Density. A surface response model was also developed for bulk density as a

function of the spray drying process variables. A complete second-order model (1) was tested for its ability to describe the response surface. Analysis of variance (Table 2) showed that the model was not significant ($P < 0.05$). In this case only β_0 was significant. Values greater than 0.1000 indicate that the model terms are not significant. The lack-of-fit P value of 0.504 implies that it is not significant. Thus, it means that none of the quadratic terms were significant. However, the determination coefficient was at 0.82.

Figures 3(a) and 3(b) show the surface and contour plots for bulk density. Figure 3(a) shows the effect of temperature and atomizer speed at the airflow centre point ($0.200 \text{ m}^3 \text{ s}^{-1}$) on bulk density. It is evident from Figure 3(a) that, at lower level of outlet temperatures and atomizer speed, the bulk density reaches the highest value, as well as higher level of airflow and low level of outlet temperature (Figure 3(b)). In Figures 3(a) and 3(b), the bulk density has decreased with the increase of the outlet temperature due to an increase in the feed flow caused by outlet temperature control in each experiment [21, 26]. This same behavior was observed for hygroscopicity, water activity, and reducing sugars.

3.5. Optimization of Spray Drying Conditions. The spray drying process was optimized to the responses using a numerical optimization technique in which an equal importance of “one” was given to all three process parameters (temperature, atomizer speed, and airflow). The process was optimized to maximize powder yield, solubility, and bulk density and to minimize hygroscopicity and water activity. As a result of, the optimum operating conditions for temperature, atomizer speed, and airflow were 80°C , 20,000 rpm, and $0.230 \text{ m}^3 \text{ s}^{-1}$, respectively. The solution for the optimum spray drying conditions was found to satisfy the goal with a powder Yield same at 73.3% w/w, solubility at 35.28 s, hygroscopicity at $1.9 \times 10^{-7} \text{ kg}_{\text{water}} \text{ s}^{-1}$, bulk density at 560 kg m^{-3} , and water activity (aw) at 0.39.

4. Conclusions

The responses were correlated with independent variables using proper transformations of the responses, and the data points were fitted in a quadratic model with significant values of R^2 . Spray drying of *Agave tequilana* juice regardless of outlet temperature, atomizer speed, and air flow did not cause a significant difference in hygroscopicity, bulk density, water activity, and reduced sugar. However, significant effects were observed for powder yield and solubility at low or high temperatures, atomizer speeds, or airflows. Spray drying of *Agave* juice at low outlet temperatures (70°C) enhanced the cloud value with a maximum powder yield. The optimization of the drying process showed that the best conditions for pilot spray drying are inlet drying temperature of 180°C and outlet drying temperature of 80°C associated with an atomizer speed of 20,000 rpm and an airflow of $0.23 \text{ m}^3 \text{ s}^{-1}$, maintaining the material at room temperature (25°C) during the feeding of the dryer. Optimization of responses was based on minimum values of water activity and hygroscopicity and maximum values of yield, solubility, and bulk density.

Nomenclature

Y: Predicted response
X: Independent variables.

Greek Letters

β_0 : Constant (intercept)
 β_i : Linear coefficient
 β_{ii} : Quadratic coefficient
 β_{ij} : Cross-product coefficient.

Subscripts

i : Number of independent variables.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

The authors would like to thank Agaveros Industriales de Jalisco, S. P. R. de R. L., for providing the *Agave tequilana* juice samples. They would like to thank America Alimentos, S. A. de C. V., for providing the whey protein isolated.

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