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Spray-Drying Hen Eggs: Effects of the Egg Yolk to Egg White Ratio and Sucrose Addition on the Physicochemical, Functional, and Nutritional Properties of Dried Products and on Their Amino Acid Profiles

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Spray-Drying Hen Eggs: Effects of the Egg Yolk to Egg White Ratio and Sucrose Addition on the Physicochemical, Functional, and Nutritional Properties of Dried Products and on Their Amino Acid Profiles

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Abstract: Manufactured egg powders can be formulated to produce food products that vary in their properties. The present study aims to determine the effect of egg white content on the physicochemical characteristics and on the functionality and nutritional value of dried whole egg (WE), egg white (W), and an egg yolk/white mixture in a 1:3 ratio (M1:3). These fresh egg products were spray-dried using sucrose—an agent recognized for its effect of protecting the protein in the egg during the drying process. Experiments were conducted in a laboratory-scale spray dryer, operated under controlled conditions, with an air inlet temperature of 120 °C. In the dried products, water activity, water solubility index, color, and pH were affected significantly as a function of the fresh egg component ratio and the added sucrose. The wettability and dispersibility in water of egg powder were improved when sucrose was added to the WE. The water-holding capacity was highest in dried egg white, and higher in the M1:3 mixture without sucrose added than in the WE with 5% sucrose. The results suggest that modifying the ratio of fresh egg yolk to egg white could lend some control over the protein and fat contents of dried egg products and over their functional properties.

Keywords: spray-drying; egg powders; sucrose; functional properties; amino acid profile

1. Introduction

Powdered egg offers an impressive versatility of use in many different products. It also facilitates handling and dosage, saves on labor, has higher microbiological stability, eases commerce and distribution, and increases the palatability of final products [1]. Rehydrated dried egg products exhibit a range of technological properties [2–7]. These functional properties of rehydrated egg, such as emulsifying capacity, foam formation, palatability, and digestibility, are strongly dependent on the physical properties of egg powders, which are able to be modified in different ways to reflect the needs of users [6,7]. Yolk and whole-egg powders are traditionally used for the preparation of omelets, scrambled eggs, mayonnaise, and sauces, while egg-white powders are employed in the preparation of meringues, desserts, and sausages, among other uses [3–6,8,9].

Egg products are characterized by their heat sensitivity during processing. The process of egg dehydration produces partial denaturalization and protein aggregation. When proteins are exposed to high process temperatures for long periods, they show a loss of color and modification of their functional properties [7]. With this in mind, evaluated the effect of moderated spray drying conditions on the functional properties of egg white and whole egg, finding that drying temperatures between 110 and 125 °C greatly preserved such characteristics as water retention capacity, foaming capacity and emulsifying ability.
The presence in dried egg powders of compounds such as superficial fat and hydrophobic groups of proteins hinders rapid rehydration. Mixing liquid egg products with hygroscopic ingredients—such as carbohydrates—is considered a suitable strategy to favor the reconstitution of the powder in water, improve its fluidity, and maintain its functional properties. Other additives such as salt, maltodextrin, triethyl citrate, silicon dioxide, and sodium silicoaluminate are used to improve the properties of the powdered product [7,10].

Moreover, the addition of sucrose to fresh eggs prior to spray-drying has been found to favor their stability and delay the protein aggregation resulting from severe heat treatments involved in the process of drying. Sucrose influences the egg powder’s physicochemical and functional properties, maintains foaming capacity, improves gelling, preserves powder emulsification capacity, prevents loss of viscosity, and hinders the Maillard reaction since it is a non-reducing sugar [6,7,11–13]. The addition of sucrose to liquid eggs favors the stability of low-density lipoproteins and improves powder rehydration, allowing the efficient migration of water into the powder, enhancing fluidity, ease of mixing with other ingredients, and the aqueous dispersibility of the powder [14,15]. Furthermore, water activity decreases with the addition of sucrose [11,12,14].

Industrially, the pH of fresh whole egg products is closely controlled prior to spray-drying, so as to achieve a pH of below 10 in the rehydrated powder. This is performed to avoid a reduction in protein solubility and the production of not only volatile sulfur compounds during heating but also antinutritional compounds such as lysine-alanine, which affects the functional, sensory, and nutritional properties of egg powders [7,16].

The factors that most influence the functional properties of the final dried powder during spray-drying are the type of atomizer, drying temperature, and feed flow [3,4,8,10,17,18]. The aqueous dilution of fresh eggs that are fed to the dryer avoids the problems derived from high viscosity, such as low circulation, nozzle plugging, and the excessive accumulation of agglomerated dried drops, which form a layer of product in the drying chamber. Although such dilution increases the time and energy consumption needed to evaporate water from the mixture, it has been shown experimentally that this drying process is more efficient as it obtains increases in the yield of the dried product [8,19,20].

This research examined the effect of the ratio of egg yolk to egg white, in liquid mixtures with added sucrose and adjustments in the pH, on the physicochemical, functional, and nutritional characteristics of spray-dried egg powders, together with their corresponding amino acid profiles.

2. Materials and Methods

2.1. Materials and Preparation of Liquid Mixtures with Egg Components

Fresh eggs with weights ranging from 67.0 to 77.9 g, sucrose from sugarcane (table sugar), and citric acid (E330) were used, and all ingredients were purchased in local markets. The egg mixtures tested were an egg yolk/egg white liquid in the same proportions as are present in fresh whole eggs, with approximately 60–68% egg white (WE); an egg yolk/white mixture (M1:3) in a 1:3 ratio, thus containing 75% egg white; and a mixture with 100% egg white (W). The sucrose addition ranged between 0 and 7.5%, the maximum level of which was set from previous experiments and the maximum content of sugar specified for the final dried egg products. The pH of WE, M1:3, and W were each homogenized for 10 min, using a three-blade mechanical agitator (IKA RW 20 Digital, Wilmington, NC, USA) at 600 rpm. After mixing, they were filtrated for the retention of aggregates larger than 250 µm. Each sample of liquid egg to be processed in the dryer was diluted with distilled water, in a ratio of liquid mixture to water of 75:25. Sucrose was added according to the ratio specified in the experimental design treatments, and all the mixtures were finally homogenized for 10 min at 700 rpm. The pH was adjusted by acidification to maintain it below 10 during processing. The pH of each liquid mixture was tested and adjusted with 23% (w/w) citric acid solution to bring the W and M1:3 to a pH of 8.2 ± 0.2 and the WE to a pH of 7.2 ± 0.2.
2.2. Characteristics of the Spray-Drying System

A Buchi-B290 Mini spray dryer, with a two-fluid spraying nozzle and nozzle tip with a 0.7 mm diameter hole, was used. The maximum inlet temperature in the drying air was able to be set at 220 °C; the gas sprayed, either compressed air or nitrogen, with controllable flows between 200 and 800 L/h, at a pressure of 5–8 bars; the flow of drying air was likewise able to be set to a maximum of 35 m³/h; the range in droplet size was set from 1–25 µm; heating control precision was set at ±3 °C, and water evaporation capacity at 1 L/h.

2.3. Drying Process Conditions

In each experimental run, 200 mL of unpasteurized liquid egg mixture, at room temperature (23–26 °C), was fed into the dryer, under constant stirring, at 200 rpm. The air pressure at the compressor outlet was held at 6.21 bars; the relative humidity of the drying air was in the range of 53–70%; the actual flow of the spraying air was set at 831 L/h; the pump with which the liquid egg dispersions were fed was set at 10% (the feeding flows for each experimental egg mixture are recorded in Table 1); inlet air temperature was set at 120 °C; the resulting outlet air temperature was in the range of 74–77 °C when W and WE egg mixtures were dried, while for the M1:3 egg mixtures, the outlet air temperature was set at 71–77 °C. The dried products in the collection vessel were weighed to evaluate the production yield of the final products, and the dried samples were then stored in hermetically sealed bags prior to analysis.

Table 1. Total solids and flows of mixtures prepared with fresh egg components and fed into the drying system (mean ± standard deviation, n = 3).

| Liquid Egg Components (Factor X₁) | Sucrose Content (%) (Factor X₂) | Total Solids (% w/w) | Liquid Flows (mL/min) |
|-----------------------------------|---------------------------------|----------------------|----------------------|
| Mixture of 100% egg white, [W]    | 0.0                             | 10.60 ± 0.03 E       | 3.25 ± 0.07          |
|                                   | 5.0                             | 14.35 ± 0.02 E       | 3.13 ± 0.08          |
|                                   | 7.5                             | 16.37 ± 0.01 D       | 3.00 ± 0.03          |
| Mixture of egg yolk: egg white (1:3), [M1:3] | 0.0                             | 13.69 ± 0.04 E       | 3.20 ± 0.04          |
|                                   | 5.0                             | 19.20 ± 0.40 C       | 3.11 ± 0.01          |
|                                   | 7.5                             | 21.70 ± 0.30 B       | 3.00 ± 0.03          |
| Mixture of components in a fresh whole egg, [WE] | 0.0                             | 18.70 ± 0.20 C       | 3.36 ± 0.02          |
|                                   | 5.0                             | 22.11 ± 0.02 B       | 3.26 ± 0.02          |
|                                   | 7.5                             | 22.92 ± 0.00 A       | 3.09 ± 0.01          |

Data are the means of three repetitions. Means of the total solids that do not share a letter are significantly different (Tukey test, p-value < 0.05).

2.4. Physicochemical Analysis of Products

Moisture content was analyzed in accordance with method 17.007-1984 of the AOAC, with certain modifications [9]; water activity (Aw) was measured at 25 °C, using the Aqualab VSA (Meter, Pullman, WA, USA); color was measured using the CM-5 spectrometer (Konica Minolta, Tokyo, Japan); pH was determined by following the methodology described in ISO 1842:1991, using the Orion Star A215 pH meter (Fisher Scientific, Loughborough, UK); total fat content was determined via the acid hydrolysis methodology, based on AOAC 922.06-1922; total protein content, in accordance with the Kjeldahl method, was based on ISO 1871:2009; and cholesterol content was determined by gas chromatography, in accordance with the AOAC 994.10-1994.

2.5. Functional Properties Analysis

Water-holding capacity (WHC, mL of water/g of wet sample) in the dried powdered egg products was determined at 25 °C, in accordance with the procedure of rehydration and centrifugation reported by Diniz and Martin [21] and Sze, et al. [22], with certain modifications in the powder to water ratio and in the speed of centrifugation; product dispersibility(s) was measured at 25 °C and 50 °C, in accordance with the method of
rehydration of powders used by Zhao, et al. [23] with modification in the speed of agitation; wettability(s) was determined at 25 °C and 50 °C following the methodology of powder rehydration reported by Ojo, et al. [24], with modifications in relation to the sample holder type for testing; WSI [%, w/w, dry basis] was evaluated at 25 °C and 50 °C using the procedure described by Anderson, et al. [25], with a number of modifications. All properties were measured in triplicate.

2.6. Amino Acid Analysis by Gas Chromatography Coupled to a Mass Detector (GC/MS)

The measurement of amino acids in the dried powdered egg products was carried out by gas chromatography using a device coupled to a mass detector (GC/MS) after derivatization. Samples and analytes were injected in the splitless mode in a gas chromatographic system (GC) Agilent 7890 (Agilent, Wilmington, DE, USA) equipped with a mass spectrometer detector (MS) 5975C and a Phenomenex Zebron ZB-5 ms column (30 m × 0.25 mm × 0.25 µm). Samples of dried powdered egg (50 mg) were taken in duplicate to measure the free amino acids via extraction with acetone, according to the methodology proposed by Restrepo-Osorio [26] for protein precipitation prior to the quantification of free amino acids, with a modification in the replacement of the precipitation agent, which was acetone; the measurement of total amino acids was carried out with 100 mg samples in duplicate, which were subjected to acid hydrolysis as reported by Llames and Fontane [27], with some modifications.

2.7. Experimental Design and Statistical Analysis

The effect of the addition of sucrose (from sugarcane) on the physicochemical properties was evaluated in the dried egg powders prepared from 100% egg white, W, the M1:3 mixture, with 75% egg white, and the egg yolk and egg white WE mixture with approximately 60–68% egg white, as found in whole eggs. The dried powders were obtained under the previously specified drying conditions. A completely randomized factorial experimental design was developed, with factor X₁ as the liquid egg component mixture, with three levels: W, M1:3, and WE. Factor X₂ was sucrose in the liquid egg mixture (%, w/w, wet basis), with three levels at 0%, 5%, and 7.5% as the independent variables. Each treatment was repeated twice, for a total of 18 runs. The response variables were as follows: moisture content (%), Aw, WSI (%), pH of the reconstituted powder, and color, which were determined by duplication. A two-way ANOVA was used. The data did not undergo any treatment before processing by the ANOVA. Raw data were used for the analysis. A parametric test of difference was selected after checking the assumption of the normal distribution of data by a Shapiro–Wilk test. Data were analyzed in relation to the experimental factors using the post-ANOVA Tukey test. The statistical data analysis used IBM SPSS software, version 26, from 2019.

3. Results

3.1. Effect of Egg Component Mixtures and Sucrose Addition on the Flow of Fresh Egg Products and on the Physicochemical Characteristics of the Dried Powders

Liquid egg flow (mL/min) and total solids (%, w/w, wet basis) in the liquid egg components were determined and are shown in Table 1. Flow values decreased as the total solids content increased. The WE mixture comprising whole egg components exhibited higher flows, both without and with added sucrose, than the W egg white liquid and M1:3 egg mixture. These results match those reported in Colombian Technical Standard NTC 6116—Food Industries, Egg Products [16], in which the whole fresh egg has a concentration of total solids in the range of 18.7–25%, higher than in egg whites (10–13.5% of total solids). This is due to the higher total solids content in egg yolk.

The total solids content in the fresh egg mixtures displayed statistically significant differences. For a constant sucrose content, total solids in the mixtures varied in the following order: W < M1:3 < WE. Total solids increased as the proportion of egg yolk was increased in the fresh egg mixtures, since the total solids in egg yolks are from 40% to 45%
Moreover, the addition of sucrose incremented the total solids, generating statistically significant differences.

The pH was adjusted by acidification of the experimental liquid egg mixtures. The resulting values after pH adjustment were $8.2 \pm 0.2$ for samples W and M1:3, and $7.2 \pm 0.2$ for sample WE, prior to spray-drying. The acidification of the mixtures controlled the pH in the rehydrated egg powders at values below 10. The results of pH tests in the fresh liquid samples and in the rehydrated powders are shown in Table 2. The addition of 5% and 7.5% sucrose resulted in a drop in pH in their powders, a modification that was not observed with pH values in the experimental treatments having no addition of sucrose. The control and adjustment of pH is a routine process carried out in those industries in which dried eggs are manufactured to prevent excessive increases in pH in the reconstituted powder, so as to keep the levels under the maximum limit set in Colombian Technical Standard NTC 6116—Food Industries, Egg Products, which is based on international standards. Therein, quality requirements are established for those egg products (from hen eggs) used as ingredients in many processed food products. In this study, the pH values of the egg powders fall within the limits set by the Colombian Technical Standard NTC 6116, in the range of 7.0–9.5 for whole eggs, and 8.0–10.0 for egg whites. The pH values within both of these ranges do not exceed pH 10, as Bhandari, et al. [7] have recommended for egg powders.

Table 2. The behavior of egg sample pH before and after drying.

| Sample                          | Sucrose Content in Fresh Liquid Sample (%) | pH in Fresh Liquid Sample | Adjusted pH in Fresh Liquid Sample | pH in Rehydrated Powder |
|--------------------------------|------------------------------------------|---------------------------|-----------------------------------|-------------------------|
| Mixture of 100% egg white, [W] | 0.0                                      | 9.23 ± 0.06               | 8.06 ± 0.08                       | 9.78 ± 0.08 A           |
|                                | 5.0                                      | 9.21 ± 0.06               | 8.08 ± 0.03                       | 9.29 ± 0.05 AB          |
|                                | 7.5                                      | 9.15 ± 0.02               | 8.10 ± 0.10                       | 9.29 ± 0.27 AB          |
| Mixture of egg yolk: egg white (1:3), [M1:3] | 0.0                                      | 8.30 ± 0.20               | 8.20 ± 0.01                       | 9.25 ± 0.20 AB          |
|                                | 5.0                                      | 8.30 ± 0.20               | 8.05 ± 0.03                       | 9.09 ± 0.08 B           |
|                                | 7.5                                      | 8.30 ± 0.10               | 8.25 ± 0.01                       | 9.24 ± 0.09 B           |
| Mixture of liquid components in a whole egg, [WE] | 0.0                                      | 7.81 ± 0.04               | 7.20 ± 0.09                       | 8.51 ± 0.12 C          |
|                                | 5.0                                      | 7.80 ± 0.10               | 7.22 ± 0.06                       | 8.44 ± 0.09 C           |
|                                | 7.5                                      | 7.70 ± 0.00               | 7.21 ± 0.03                       | 8.50 ± 0.07 C           |

Means of pH of rehydrated powder that do not share a letter are significantly different (Tukey Test, $p$-value < 0.05).

On comparing the pH in the egg fresh product with that in the egg product reconstituted from powder, when using a one-way ANOVA, a significant difference was found between the products ($p$-value = 0.029). During the drying process, the heat exposure of basic branched-chain amino acids and the loss of CO$_2$ from the liquid product could lead to a pH increment in values up to 10 or higher if it is not adjusted by acidification of the fresh product previous to the drying process. Table 2 shows the pH adjusted in fresh egg samples to comply with Colombian Technical Standard NTC 6116 [16].

The effect on the physicochemical properties of dried egg products due to adding sucrose to the liquid components in eggs was evaluated from moisture content (MC, % w/w, wet basis), A$_w$, pH, and WSI (%, w/w) measurements for each experimental treatment (see Table 3). The A$_w$ in the dried egg products was significantly influenced by the sucrose added to the fresh egg mixtures ($p$-value = 0.001). Koç, et al. [11] observed that the addition of carbohydrates to fresh eggs before drying can modify water activity due to alterations in the structure and the superficial area of the egg powder. The pH in the reconstituted egg powders was influenced by the type of egg component mixture and by the sucrose added. As the amount of egg white was increased in the mixture, there was an increase in the pH of the reconstituted egg powder, which can be explained by the alkaline nature of the egg white [8]. In contrast, with the addition of sucrose, there was a decrease in the pH of the reconstituted egg powders ($p$-value = 0.036). The effect of the addition of sucrose on the water solubility (WSI) of the egg powders depended on the type of egg component mixture.
(p-value = 0.01, for the interaction between sucrose and type of mixture). The addition of carbohydrates reduces the migration of fat to the surface of the dried powder particles, favoring the humectation of the product [11], which was evident in the WE egg product with higher solubilities when 5 and 7.5% of sucrose were added, compared with the WE product with no added sucrose.

The moisture content of all of the dried egg products was below 4%. The moisture content of the W dried egg white showed a tendency toward decreasing as the addition of sucrose was increased, but for the dried egg mixtures M1:3 and WE, higher moisture contents were observed when the sucrose concentration was 5%. The Aw value in all the experimental treatments was below 0.4, a generally accepted level, to ensure the stability of the whole egg components in powder form [9]. The dried egg products with 5% sucrose added had the highest Aw values. The WSI values were over 80%, and the highest values were observed in the M1:3 egg mixture, with a maximum of 92.96 ± 1.63% (with no sucrose added) (see Table 3). Dried WE components had the lowest WSI of 83.15 ± 0.94% (with sucrose concentrations at 0%), which increased to 90.38 ± 0.68% when the sucrose content was increased to 5%. The color was evaluated by the CIELAB chromaticity model (referred to here as L*, a*, and b*). In accordance with the results in Table 3, where L* represents the value of lightness, defined from 0 (black) to 100 (white), egg component W (egg white) had the highest values, while the lowest values were in the WE mixture, which had the lowest egg white content. The a* and b* coordinates had relatively similar values for egg components in WE and in the M1:3 mixture, although they were slightly lower for M1:3. The b* coordinate had positive values toward yellow, which is characteristic of egg yolk. The analysis of variance (ANOVA) of the results is presented in Table 4. Factor \( X_1 \) (egg components in the experimental samples) had a statistically significant effect (\( p < 0.05 \)) on the WSI response variables, pH in reconstituted powder, and L*, a*, and b*. The \( X_2 \) (sucrose content, %) factor had a statistically significant effect (\( p < 0.05 \)) on Aw, WSI, and pH, and showed the interaction of \( X_1^* X_2 \) on the WSI variable.

Table 3. Results of the physicochemical characteristics of the dried powders of processed egg component mixtures (mean ± standard deviation).

| Liquid Egg Components (Factor \( X_1 \)) | Sucrose Content (% w/w) (Factor \( X_2 \)) | MC (% w/w) | WSI (% w/w) | Aw | Color |
|----------------------------------------|------------------------------------------|------------|-------------|----|-------|
| Mixture of 100% egg white, [W]         | 0.0                                      | 3.76 ± 0.09 A | 88.63 ± 0.27 B | 0.196 ± 0.002 B | L* A = 95.6 ± 0.2, a* C = −0.6 ± 0.2, b* B = 8.0 ± 1.2 |
|                                        | 5.0                                      | 3.42 ± 0.40 A | 88.46 ± 0.12 AB | 0.290 ± 0.008 A | L* A = 95.5 ± 0.4, a* C = −0.5 ± 0.1, b* B = 6.1 ± 0.5 |
|                                        | 7.5                                      | 2.11 ± 0.42 A | 88.20 ± 0.54 B | 0.235 ± 0.034 B | L* A = 95.5 ± 0.5, a* C = −0.6 ± 0.0, b* B = 6.8 ± 0.7 |
| Mixture of egg yolk: egg white (1:3) [M1:3] | 0.0                                      | 2.03 ± 0.97 B | 92.96 ± 1.63 A | 0.231 ± 0.006 B | L* B = 88.6 ± 0.1, a* B = 5.6 ± 0.4, b* B = 21.7 ± 0.6 |
|                                        | 5.0                                      | 2.72 ± 0.54 B | 90.91 ± 1.64 AB | 0.277 ± 0.008 A | L* B = 89.4 ± 0.1, a* B = 4.9 ± 0.4, b* B = 18.6 ± 0.8 |
|                                        | 7.5                                      | 2.15 ± 0.63 B | 90.88 ± 1.13 AB | 0.247 ± 0.028 B | L* B = 90.4 ± 1.7, a* B = 5.4 ± 1.7, b* B = 18.7 ± 2.1 |
| Mixture of liquid components in a whole egg [WE] | 0.0                                      | 2.22 ± 0.24 AB | 83.15 ± 0.94 C | 0.248 ± 0.002 B | L* B = 86.7 ± 1.8, a* B = 7.8 ± 1.0, b* B = 26.3 ± 5.4 |
|                                        | 5.0                                      | 2.53 ± 0.28 AB | 90.38 ± 0.68 AB | 0.271 ± 0.001 A | L* B = 86.9 ± 0.3, a* B = 6.0 ± 0.0, b* B = 22.9 ± 3.0 |
|                                        | 7.5                                      | 2.21 ± 0.26 AB | 88.47 ± 0.36 B | 0.238 ± 0.013 B | L* B = 89.3 ± 0.3, a* B = 5.8 ± 0.2, b* B = 20.7 ± 1.0 |

MC = moisture content, WSI = water solubility index, Aw = water activity, \( L^* \) = lightness value, \( a^* \) = value of chromaticity coordinate \( a^* \), and \( b^* \) = value of chromaticity coordinate \( b^* \) in the CIELAB color space model. Data are the means of two repetitions of each experimental treatment and two replicates of the analysis of each dried powder sample. Means that do not share a letter are significantly different (Tukey test, \( p \)-value < 0.05).
### Table 4. Results of the ANOVA variance analysis for the physicochemical properties of liquid egg components that are processed into dried powders.

| Variable    | Model | DF | X₁ | X₂ | X₁*X₂ | Error | Total |
|-------------|-------|----|----|----|-------|-------|-------|
| MC [%]      | SS    | 6.21 | 2.47 | 1.73 | 2.02 | 2.17 | 8.38 |
|             | p-value | 0.05 A | 0.03 A | 0.07 | 0.17 |       |       |
| Aw          | SS    | 0.01 A | 0.00 A | 0.01 A | 0.00 A | 0.02 A |       |
|             | p-value | 0.01 A | 0.41 | 0.00 A | 0.10 |       |       |
| WSI [%]     | SS    | 119.45 | 56.04 | 12.04 | 51.38 | 8.47 | 127.92 |
|             | p-value | 0.00 A | 0.00 A | 0.02 A | 0.00 A |       |       |
| pH          | SS    | 3.34 | 3.00 | 0.18 | 0.17 | 0.16 | 3.51 |
|             | p-value | 0.00 | 0.00 | 0.04 | 0.14 |       |       |
| Lightness (L) | SS   | 193.43 | 182.54 | 6.10 | 4.78 | 7.02 | 200.45 |
|             | p-value | 0.00 | 0.00 | 0.06 | 0.27 |       |       |
| Chromaticity coordinate a* | SS   | 176.39 | 171.08 | 2.31 | 3.01 | 4.37 | 180.76 |
|             | p-value | 0.00 | 0.00 | 0.15 | 0.27 |       |       |
| Chromaticity coordinate b* | SS   | 930.07 | 882.44 | 37.01 | 10.62 | 46.20 | 976.27 |
|             | p-value | 0.00 | 0.00 | 0.07 | 0.73 |       |       |

MC = moisture content, Aw = water activity, WSI = water solubility index, DF = degree of freedom, SS = type III sum of squares, X₁ = liquid egg component mixture, X₂ = sucrose content (%). A Statistically significant differences at p-value < 0.05.

#### 3.2. Drying Yields and Nutritional Compositions of Selected Egg Component Powder

The W and M1:3 egg products without sucrose, as well as the WE with 5% sucrose added (WE5%), were selected as those egg powders with the best reconstitution properties due to their high WSIIs, which is a favorable characteristic in food manufacture. Following the selection of these egg products, additional drying experiments were conducted in which the yields of dried products were determined. For these additional experiments, six lots were processed, using each experimental liquid egg preparation: W, M1:3, and WE5%, with the same drying conditions as previously specified (see Section 2.3). The mean yield results for drying tests with W, M1:3, and WE5% were 82 ± 2%, 91.2 ± 0.6%, and 91 ± 1%, respectively. The six examples of each egg product, in powder form, were mixed in polypropylene bags and stored in metalized, sealed bags at 5 ± 1 ºC. Samples from each dried egg product were used to evaluate nutritional compositions, as reported in Table 5.

The results in Table 5 indicate that egg powder W has the highest content of protein and ash and the lowest content of lipids, cholesterol, and calories. The highest content of moisture and carbohydrates corresponded to the powder WE5%.

### Table 5. Mean values for the nutritional compositions of egg powders W, M1:3, and WE5%.

| Analysis                      | W     | M1:3  | WE5%  |
|-------------------------------|-------|-------|-------|
| Moisture and volatile materials (g/100 g) | 4.51 | 3.22 | 4.79 |
| Total protein (g/100 g)       | 80.48 | 58.55 | 39.92 |
| Total fat (g/100 g)           | 1.21 | 29.61 | 13.18 |
| Total carbohydrates (g/100 g) | 8.19 | 4.45 | 39.42 |
| Ash (g/100 g)                 | 5.61 | 4.17 | 2.69 |
| Cholesterol (mg/100 g)        | 5.62 | 553.41 | 518.36 |
| Total calories (kCal/100 g)   | 365.57 | 518.49 | 435.98 |

W = egg white, M1:3 = a mixture of 1 part egg yolk and 3 parts egg white, and WE5% = whole egg with the addition of 5% sucrose. The results are reported on a wet basis.

#### 3.3. Functional Properties of Powdered Egg Products W, M1:3, and WE5%

In order to evaluate the functional properties of the W, M1:3, and WE5% dried egg products, WHC at 25 ºC and WSI, dispersibility in water, and wettability at 25 ºC and 50 ºC were determined. The W egg product had the highest WHC value, 3.03 ± 0.00 mL water/g of powder, and the lowest ISA value, at 25 ºC and 50 ºC, which agrees with the observation...
made by Diniz and Martin [21] that whenever a product has a high WHC, it also exhibits a low WSI (see Table 6).

Table 6. Values for the functional properties of egg powders C, M1:3, and WE5% (means ± standard deviation).

| Functional Property | Temperature (˚C) | W        | M1:3     | WE5%     |
|---------------------|------------------|----------|----------|----------|
| WHC (mL water/g wet sample) | 25               | 3.03 ± 0.00 | 0.91 ± 0.00 | 0.30 ± 0.00 |
| WSI (%)             | 25               | 88.63 ± 0.27 | 92.96 ± 1.63 | 90.38 ± 0.68 |
|                     | 50               | 76.64 ± 1.05 | 90.97 ± 0.93 | 82.76 ± 0.35 |
| Dispersibility (s)  | 25               | 3440 ± 183 | 530 ± 1   | 360 ± 4   |
|                     | 50               | 372 ± 2   | 163 ± 4  | 84 ± 4   |
| Wettability (s)     | 25               | 4700 ± 92 | 5443 ± 41 | 474 ± 7  |
|                     | 50               | 7478 ± 21 | 3869 ± 40 | 118 ± 2  |

W = egg white, M1:3 = a mixture of 1 part egg yolk and 3 parts egg white, and WE5% = whole egg with the addition of 5% of sucrose. WHC = water-holding capacity, WSI = water solubility index.

3.4. Amino Acid Profile of Powdered Egg Products WE5%, M1:3, and W

Figures 1 and 2 present the profiles of free and total amino acids in the WE5%, M1:3, and W egg powders. In Figure 1, it can be seen that the WE5% sample had the highest concentration of the free amino acids and essential amino acids analyzed: L-isoleucine, L-leucine, L-lysine, L-methionine, L-phenylalanine, L-threonine, L-tryptophan and L-valine, followed by sample M1:3. In contrast, for total amino acids, Figure 2 shows that in terms of the six essential amino acids analyzed, sample M1:3 had the highest content of L-lysine, L-phenylalanine, and L-threonine, sample W the highest content of L-leucine and L-valine, and sample WE5% the highest content of L-isoleucine.

![Free amino acids](image_url)

**Figure 1.** Free amino acid composition of egg powders: whole egg with the addition of 5% sucrose (WE5%); a mixture of 1 part egg yolk and 3 parts egg white (M1:3); and egg white (W). Average data (n = 2). * Essential amino acids. Bars represent the standard deviation.
4. Discussion

4.1. The Physicochemical Properties of Egg Powders

The effect of the egg yolk to egg white ratio and the addition of sucrose (0%, 5%, and 7.5%), on the physicochemical properties of the dried products obtained by spray-drying was evaluated.

The production yields of the dried egg products W, M1:3, and WE5% equated to values of 82 ± 2%, 91.2 ± 0.6%, and 91 ± 1%, respectively. These results are probably due to the air temperatures at the dryer chamber outlet, which were close to the glass transition temperatures reported for whole eggs (59–63 °C) and egg whites (98–115 °C). The outlet air temperature was in the range of 74–77 °C during the drying of W and WE, and in the range of 71–77 °C during the drying of M1:3, conditions that prevented the development of high material stickiness and, consequently, relatively high production yields [7,28–30].

The dried egg products had Aw values lower than 0.3, due to their low moisture contents (below 3.8%) and high compositions of protein and carbohydrates, molecules with a high capacity for binding with water. The W egg product with 0% sucrose had the lowest Aw (0.196 ± 0.002) and the highest protein content (80.48%), binding higher amounts of water than do carbohydrates (see Tables 3 and 5) [30]. The low water activity of the egg powder products favors stability during storage. Optimal stability is usually achieved for Aw at between 0.2 and 0.3 [30], which was the range of the Aw in the products obtained in this research. Furthermore, these values of water activities were similar to those reported by Schuck, et al. [30] for the whole egg, egg yolk, and egg white (0.25, 0.25, and 0.23, respectively).
In accordance with the results in Table 3, WE showed an increase of 8.7% in WSI value at 25 °C when 5% of sucrose was added [14]. In contrast, W and M1:3 showed no WSI increase when sucrose was added. Some of the reasons for this behavior include high protein content in these egg products (see Table 4), as it is believed that the higher protein content in egg powders causes reduced dispersing capacities in water when proteins absorb water and swell, and this phenomenon reduces the ability of the material to progressively disintegrate into particles of smaller sizes [30]. However, under the process conditions studied, WSI values of above 80% were obtained for all powdered egg products. These were higher than those reported by García-Figueroa [10] for powdered whole egg, processed in a spray-dryer set with a disk rotating at high speeds to spray the feed liquid. García-Figueroa [10] studied a dried mixture composed of 87% unpasteurized liquid whole egg, 12.7% maltodextrin DE 17–20, and 0.33% salt, dried at 180 °C. The WSI value of the dried product was 75.5 ± 0.5%. This could be due to the effect of drying temperatures, the dissolution in water of the liquid mixture, mixture compositions, or particle size, which is dependent on the atomization system and affects the dissolution rate. This is faster with smaller particles because the surface area increases as particle size decreases [8,19,30].

The value of lightness and coordinates a* and b* produced numerous variations compared to those reported by Schuck, et al. [30]. This could be due to the specific characteristics of the feed material and the moderate drying air-temperature conditions (120 °C) used in this investigation. These could have triggered the color change to brown and caused pigment oxidation [11]. In egg powders, the color results depend on the chemical compositions of liquid eggs, thus influencing liquid color, which may vary from very light yellow to a vivid, dark orange. The carotenoids in egg yolks can experience structural changes during spray-drying, due to the high processing temperatures and airflows, which could cause the activation of oxidative reactions and, consequently, change the color of egg powders. Additionally, the presence of reducing sugars and proteins in the chemical composition of liquid eggs can activate browning reactions [11].

4.2. The Nutritional Composition of Dried Egg Components W, M1:3, and WE5%

From the WSI results shown in Table 3, three mixtures of egg powder with high WSI values were selected (W, M1:3, and WE5%) due to their ease of reconstitution in water. The nutritional composition and functional properties of the selected products were analyzed. In accordance with the results reported in Table 5, moisture content fell below those limits established by the Colombian Technical Standard NTC 6116 for dried egg whites and dried whole eggs, with values of ≤ 8% and ≤ 5%, respectively [16].

According to the results in Table 5, protein content was highest in egg component W, at 80.48 g/100 g, close to the 79.34 g/100 g value reported by Ayadi, et al. [8] at drying temperatures in the range of 110–125 °C, and with a feeding flow of 3.30–5.00 mL/min, these conditions being quite similar to those used for drying egg whites in this investigation. Egg product W has a higher protein content than egg products M1:3 and WE5% because W is composed of egg white, which contains a high proportion of ovalbumin (54%) and other ovoproteins [31]. Additionally, the W egg component meets the bromatological requirements specified for protein content in the Colombian Technical Standard NTC 6116, or 78.0–83.0 g/100 g [16].

The WE5% egg product, with 39.4% carbohydrates, had a protein content of 39.92 g/100 g. This value for protein is slightly higher than that of 38.4 g/100 g, reported by OVODAN [32] for whole eggs in powder with 20% sucrose. Depending on the product application, whole egg powder with 10–40% carbohydrates may be found available commercially [14]. The highest carbohydrate content was in WE5%, with a value of 39.42 g/100 g sample, which is due to the added sucrose. The values for W and M1:3 were 8.19 g/100 g and 4.45 g/100 g, respectively, neither of which had added sucrose. The carbohydrate content in these egg products is represented principally by the free glucose found in the liquid egg white (0.34 g/100 g), although traces of fructose, lactose, maltose, and galactose were also detected in egg whites and yolks [33,34], which were found in lower proportions in M1:3.
M1:3 had the highest fat content, with a value of 29.61 g/100 g, and the lowest carbohydrate content, with an average value of 4.45 g/100 g. This result for fat content is considered low compared to 36.1 g of fat/100 g for dried whole egg, as reported by Schuck, et al. [30], and 39.2 g/100 g, as reported by Froning [35], which can be explained because the experimental liquid egg M1:3 contained 25% of egg yolk, while liquid whole egg contained 32–40%, fat being the main component of egg yolk solids. The egg product WE5% contained 13.18 g/100 g of fat, 39.92 g/100 g of protein, and the highest carbohydrate content at a value of 39.42 g/100 g, which explains the lower proportion of fat compared to that in egg product M1:3. Ayadi, et al. [8] reported traces of fat in egg white powders, which accounts for the low content of fat in egg product W, with a value of 1.21 g/100 g. The cholesterol content found in WE5%, M1:3, and W was 518.36 mg/100 g, 553.41 mg/100 g, and 5.62 mg/100 g, respectively. Many years ago, the cholesterol contained in egg yolks was concerning, due to cholesterol’s association with specific health problems. However, more recently, studies have indicated that dietary cholesterol has only a limited effect on those health problems [33,36].

The highest ash content was found in W, with a value of 5.61 g/100 g, in agreement with that reported by Ayadi, et al. [8], of 5.69 g/100 g. The ash value found in M1:3 was 4.17 g/100 g, and, in WE5%, 2.69 g/100 g. The latter value is inferior to that reported for whole eggs by Ayadi, et al. [8], of 6.13 g/100 g, and Froning’s [35] of 3.4 g/100 g, which is due, of course, to the sucrose content in WE5%, which affects the relative mineral concentrations. Additionally, the mineral composition in eggs may be affected by the type of feed, the poultry-raising system used, and the genetic characteristics of the hens [37].

4.3. Functional Properties, as Affected by Types of Egg Components in W, M1:3, and WE5%

According to the data in Table 6, the greatest water-holding capacity was found in the W dried egg component with a value of 3.03 mL of water/g, which is higher than the value reported of 2.58 mL water/g for egg whites dried via freeze-drying [38]. A high WHC value can indicate a high degree of protein denaturation in egg white, which is particularly thermolabile. Ovotransferrin (which represents approximately 12% of egg white albumen) is one of the most thermosensitive proteins, with a denaturation temperature of just 61 °C [31]. During spray-drying, proteins are subjected to denaturation phenomena and partial breakdown caused by acidification, spraying, and heat treatment. These contribute to the formation of a stable protein matrix, made up of denaturalized proteins with enhanced water retention properties [39]. The results show an inverse relationship between water-holding capacity and egg powder water solubility, which confirms the information reported by Pokora, et al. [40]. The egg components in the M1:3 mixture had the highest WSI, tested at 25 °C and 50 °C, followed by the egg components in WE5% and W. The WSI values were lower at 50 °C than at 25 °C for all dried egg products, which in the case of egg whites is in agreement with the results reported by Gomes and Pelegrine [41], who evaluated water solubility in dried egg whites in the 40–100 °C temperature range, with higher solubility found at 40 °C than at 50 °C.

In relation to dispersibility in water, the shortest time for material dispersion corresponded to WE5% at both at 25 °C and 50 °C, with dispersion times of 360 s and 84 s, respectively. The high concentration of soluble carbohydrates, 39.42 g/100, in WE5% enhanced the material’s dispersibility in water due to the kinetics of powder rehydration, which depends on their compositions and their consequent affinity for water, sucrose being the main carbohydrate in WE5%, which is highly hygroscopic [11,12,14,30]. Rehydration at 50 °C enhanced the process dynamic in all dried egg products, permitting product particle disintegration in water after shorter times. WE5%, when processed at 50 °C, could be considered a wettable material, considering that its time of wetting was less than 120 s [30]. This is due to the sucrose content in the egg product, which prevents the migration of fat and its concentration on the particle surface during spray-drying, thus favoring powder reconstitution in water because there is not a fat layer on the particle surface to impede water absorption [11]. In contrast, the times for wetting W and M1:3 were above 3800 s at
25 °C and 50 °C (see Table 4). These results confirm the report of Zeidler [42], who reported that egg whites, in flakes, require between 6 and 8 h for their reconstitution in water, which shows that the hydration of egg powders is normally difficult and takes time. According to Schuck, et al. [30], organic powders with a high content of protein or fat, as is the case with egg mixture M1:3, have wettability times of over 120 s; for this reason, they are not considered instant powders. The structural collapse caused by water diffusion during drying and the high temperatures used in the process also affect the reconstitution of dried egg powders [42].

### 4.4. pH Behavior and the Amino Acid Profile of Egg Samples

The composition of free amino acids in Figure 1 for sample W shows a higher composition of hydrophilic amino acids, L-aspartic acid 11.7 ± 0.10, L-arginine 3.94 ± 0.01 and L-glutamic acid and glutamine 3.70 ± 0.30, followed by hydrophobic amino acids, such as L-phenylalanine 3.25 ± 0.00 and L-tryptophan 1.75 ± 0.01. In the analysis of the total amino acids, the amino acid trans-4-hydroxy-L-proline was found only in sample W at a concentration of 2.56 mg/100 g (dry basis) (see Figure 2). The presence of L-arginine as a free amino acid is one of the causes of the change in pH experienced by sample W after drying, which went from a pH of 8.06 for the liquid sample to 9.78 for the dried powder. During the drying process, the L-arginine side chain, guanidine, may be exposed to heat denaturation, which causes an increase in pH due to the alkaline nature of this substance. Likewise, the exposure of groups of the side chain of other basic amino acids increases the pH of egg powders [8]. The increase in the pH of dried egg products has also been attributed to the evaporation of dissolved CO$_2$. The release of this gas produces changes in the balance of the carbonate–bicarbonate buffer present in the egg. The system tries to compensate for the loss of gas by moving the balance to the carbonate side, causing an increase in pH [8,43].

According to the results of the total amino acid composition of the egg powders (see Figure 2), the predominant amino acids for sample W were: L-valine 3638 ± 1053; L-leucine 2917 ± 741, and L-alanine 1265 ± 35, which are hydrophobic because they present nonpolar groups in their side chain, such as isopropyl, isobutyl, and methyl, respectively [44]. This could be one of the causes of the lower solubility of W compared to that in WE5% and M1:3 since the solubility of proteins in water is influenced by the pH and the functional group present in the side chain of its structure [45]. The hydrophobicity of these amino acids could be one of the causes of the long wettability times found for sample W of 4.700 s at 25 °C and 7.478 s at 50 °C. The denaturation of proteins during the spray-drying process of the egg white causes conformational changes that lead to the exposure of hydrophobic groups on its surface, thus reducing the wetting capacity of the product [46].

The addition of sucrose to the treatments with 5% and 7.5% for sample W prevented the pH from exceeding a value of 10, as established in NTC 6116 [16]. This could be explained by the protective nature of carbohydrates, which prevent the denaturation of proteins and, consequently, the exposure of the side chains of basic amino acids, such as L-arginine, L-leucine, and L-histidine [7,13].

In the WE5% sample, the predominant amino acids were L-aspartic acid and L-glutamic acid. These have carboxyl groups in their side-chain that completely ionize at neutral pH, favoring their solubilization in water [47]. This could account for the fact that the pH of the WE5% powder was lower compared to that of the W and M1:3 samples (see Table 2), which presented a lower proportion of these two amino acids. The pH of all rehydrated egg powders was far from the corresponding isoelectric point (IP) reported for the predominant amino acids in each sample: L-aspartic acid for WE5%, with a PI of 2.77; L-glutamic acid for M1:3, with a PI of 3.22 and L-valine for W, with a PI of 5.96, which could have promoted their solubility in water [47].

The contents of free amino acids in fresh eggs are influenced by chicken breeds and feed compositions. Likewise, the amount and distribution of free amino acids in egg powders influence the sensory and functional properties of food products [48]. Goto, et al. [48]...
reported that amino acids are precursors of different flavors by interacting with other components in food matrices. Amino acids, such as histidine, glycine, arginine, alanine, valine, methionine, tryptophan, phenylalanine, isoleucine, leucine, lysine, and proline have been associated with the perception of a bitter taste. Likewise, Nimalaratne, et al. [49] found that the presence of tryptophan and tyrosine in egg yolk is the main contributor to antioxidant capacity. According to the above findings, the results of free amino acids in egg powders observed in Figure 1 may have an influence not only on their nutritional and functional characteristics but also on their sensory characteristics and possible applications in the food industry.

Aspartic acid and glutamic acid are the amino acids found in the highest proportion in the WE5% mixture. This is consistent with the findings reported by Attia, et al. [50], where these amino acids were found in greater proportions for the whole egg. Glutamic acid was found in the M1:3 and WE samples since this amino acid is characteristic of egg yolk, which is found in the highest proportions in these samples [50].

Valine and leucine were the characteristic amino acids of sample W. As reported Attia, et al. [50], these amino acids are found in greater proportion in the egg white compared to the yolk and the whole egg. These amino acids are part of the characteristic proteins of egg white, such as ovalbumin, ovotransferrin, and ovomucoid [12].

It is worth noting that the valine and leucine levels were higher in WE5% than in M1:3, despite the fact that the egg white content in M1:3 is higher than in WE5%. This could indicate that the carbohydrates added to the mixture preserved these amino acids. As reported by Koç, et al. [11], carbohydrates have a protective effect on proteins by forming hydrogen bonds that promote protein stability.

Fresh and dried whole egg, egg yolk, and egg white contain all the essential and non-essential amino acids in different proportions, according to the feeding regimen of the laying hens, the freshness of the fresh and dried egg products, and the conditions of the drying process, among other factors. This shows the potential to improve nutritional values and shelf life by controlling the concentrations of essential amino acids and antioxidants in powdered egg products [50]. The standard values of the Food and Agriculture Organization (FAO) and the World Health Organization (WHO) related to the essential amino acids evaluated in this research and expressed in g/100 g of protein are: L-valine, 3.9; L-isoleucine, 3.0; L-leucine, 5.9; L-lysine, 4.5; and L-threonine, 2.3 [50]. Comparing the concentration of the essential amino acids evaluated in the egg products in this investigation (see Figure 1) with the standard values referring to pure protein from the FAO/WHO, it was found that the WE5% sample contained 34.27% of L-isoleucine, sample M1:3 contained 49.17% L-lysine and 87.97% L-threonine, and sample W contained 93.27% L-valine and 49.44% L-leucine.

5. Conclusions

This study suggests that modifying the ratio of liquid egg yolks to egg whites in mixtures prepared for spray-drying could enable greater control over protein and fat content and that of other components in dried egg products; this would allow for variance in the behavior of the materials when reconstituted in water. This would consequently permit their functional properties to be modified, thus preserving the very specific aromatic potential of egg yolk in egg products.

The addition of sucrose to the whole egg liquid components improved the reconstitution process of the dried products, as seen with the increase in the water solubility index. Adding 5% and 7.5% sucrose to egg whites, a mixture with a 1:3 (M1:3) ratio, and to the mixture of egg yolks and whites in the proportions found in whole eggs, showed a reduction in pH of the reconstituted powdered egg compared to egg products without added sucrose. This is a positive fact that may contribute to the control of pH within the limits established in the regulations for whole egg and egg white powders.

The distribution of free amino acids in dried egg samples found in this research is a nutritional characteristic that opens the door to future research to evaluate the effect of the addition of sucrose and the adjustment of pH on the amino acid profile before
and after spray-drying. In parallel, it is important to evaluate the effect of amino acid composition on the sensory properties of the dried products, as well as on their digestibility and antioxidant capacity.

The egg products tested in this research would be expected to have high stability, given their low values of water activity. These egg products are considered to have great potential for their varied nutritional and functional properties, being well-suited for their use in the formulation of foods such as sauces, meat emulsions, food pastes, confections, and especially in instant formulas for drinks, baby cereals, and all-purpose mixtures.

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