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COMMUNICATION

The Adjuvants Aerosil 200 and Gelita-Sol-P Influence on the Technological Characteristics of Spray-Dried Powders from *Passiflora edulis* var. *flavicarpa*

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ABSTRACT

Passiflora edulis *(passionflower) is a plant widely used in the Brazilian popular medicine and phytopharmaceutical industry for its sedative activity. This work refers to the development of spray-dried powders (SDPs) from the 40% ethanolic extractive solution of* P. edulis *aerial parts. The SDPs were prepared with a Büchi 190 Mini-spray dryer using as drying adjuvants Aerosil 200® alone (SDP1), an Aerosil 200 : Gelita-Sol-P® (1 : 1) mixture (SDP2) and an Aerosil 200 : Gelita-Sol-P (1 : 3) mixture (SDP3). All the powders were obtained using 40 parts adjuvant and 60 parts extract dried residue. The comparison criteria applied were particle size distribution, hygroscopicity at 95%, 60%, and 35% relative humidity (RH), as well as the flavonoid process recovery. The particle size distributions were analyzed by way of (*a*) normal distribution parameters, (*b*) the RRSB grid and (*c*) considering diameter values in terms of an equivalent sphere. All the powders presented nonnormal distribution, and the RRSB analysis appeared to be, therefore, the analysis method of choice. The total flavonoid recovery was around 80%, and it was practically not affected by the SDP1, SDP2, and SDP3 compositions. At the 60% and 90% RH atmospheres, the SDP3 and SDP2 moisture uptakes were higher than that of the SDP1. All the formulations were, on the contrary, stable at 35% RH, showing a slight moisture loss tendency. The results showed in sum that the SDP prepared using Aerosil 200 as the drying adjuvant alone presented the best technological characteristics of all.*
Key Words: *Passiflora edulis; Phytopharmaceutical technology; Spray-dried powders.*

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INTRODUCTION

Dried medicinal plant extracts have been a matter of interest of the Brazilian phytopharmaceutical industry. Recently, vegetable spray-dried extracts have been regarded as a current technological alternative in the preparation of instant soluble products or intermediary products intended for preparation of solid and semisolid dosage forms (1,2). The benefits of the utilization of spray-drying techniques on the development of plant dried extracts are mainly the preservation of thermolabile substances, if compared to other heat drying techniques, and its relative low production cost. The type and amount of the drying adjuvant influence on the quality and stability of vegetable extracts are well known in the practice (3–9).

This paper, a comparative study of the influence of Aerosil® 200 and Gelita-Sol-P® on the *Passiflora edulis* spray-dried powders (SDPs) preparation, considers the particle size distribution, hygroscopic behavior at different relative humidities (RHs), and the flavonoid process recovery as evaluation criteria.

MATERIALS AND METHODS

Extractive Solution Preparation

Passiflora edulis dried aerial parts (500 g) were extracted by turbo-extraction using an Ultra-turrax T-45 (Janke and Kunkel) over 15 min at 10,000 rpm in 5 L of 40% (v/v) ethanol.

Extractive Solution Characterization

Dry Residue

Five drug samples of 20.0 g each were analyzed, separately, according to the German Pharmacopoeia (10).

Total Flavonoid Content

The extractive solution was quantified according to the modified method of Schmidt and González Ortega (11). The total flavonoid content was expressed as grams of apigenin per 100 g of dried sample (Eq. 1). Each value represents the mean value of three determinations.

$$C = \frac{AFD}{mE_{1cm}^{1\%}}$$

where C is the total flavonoids content expressed as grams of apigenin in 100 g of sample, A is the measured absorption (U.A.), FD is a dilution factor (2500), m is the sample weight (g), and $E_{1cm}^{1\%}$ is the apigenin-$AlCl_3$ complex specific absorption.

Table 1

Percentage Composition of the Passiflora edulis *Spray-Dried Powders*

Product Designation	Drying Adjuvants (g)		Dried Residue (g)	Total Weight (g)
	Aerosil 200	Gelita-Sol-P		
SDP1	40	0	60	100
SDP2	20	20	60	100
SDP3	30	10	60	100

Spray-Dried Powder Preparation

The extractive solution was concentrated under vacuum (evaporator Büchi R-114) to about one-fourth of the original weight. The adjuvants were dispersed at room temperature using different ratios of adjuvant to dry residue mixture (Table 1). The drying adjuvants were Aerosil 200 alone (SDP1), an Aerosil 200:Gelita-Sol-P (1:1) mixture (SDP2) and an Aerosil 200:Gelita-Sol-P (1:3) mixture (SDP3). The spray-dryer conditions were 0.5-mm i.d. air nozzle, inlet temperature of 150°C; outlet temperature 90°C; 1.2 ml/min feed ratio; and air exhaustion capacity at level 4. No recycling feed system was attached to the module.

Spray-Dried Powder Characterization

Moisture Content

Each SPD was assayed as directed in the loss on drying essay monograph of the German Pharmacopoeia (10) using 300-mg samples. The results correspond to the mean value of three determinations.

Particle Size Analysis

The SPD particle size determinations were performed in accordance with Feret's diameter principle using an optical microscope calibrated with stage micrometer scale (12). The average diameters were estimated by statistical analysis assuming a normal distribution, nonnormal distribution (RRSB grid) (13) and considering diameter values in terms of an equivalent sphere (geometric method) (12), by counting of 1000 particles for each analysis.

Table 2

Comparative Evaluation of the Particle Size Parameters Determined by the Normal Distribution (M1), RRSB Grid (M2), and Equivalent Sphere (M3) Methods

	SDP1			SDP2			SDP3		
Parameters	M1	M2	M3	M1	M2	M3	M1	M2	M3
Mean diameter (μm)	68	85	91.1	72	85	95.4	53	70	70.3
Particle size distribution amplitude	−0.9	1.75	—	−0.81	1.70	—	−0.06	1.63	—
Specific surface (cm^2/cm^3)	—	1380.2	—	—	1430.5	—	—	1820	—
Diameter-specific volume (cm^3/cm^2)	—	—	137.7	—	—	138	—	—	101

Hygroscopicity Evaluation

Samples of 0.5 g were exposed to the 35%, 65%, and 95% RH atmospheres at 25°C ± 1°C. After 1, 2, 3, 4, 6, 8, 10, 12, and 15 exposition days, the sample weight differences were determined, and the respective isotherms were plotted. Each point represents the mean value of five determinations.

Total Flavonoids Content

The SPD samples, 500 mg each, were dissolved in 50 ml of 40% (v/v) ethanol, shacked for 15 min, and diluted to 100 ml with ethanol 40% (v/v). Each SDP solution was diluted in such a way that the final concentrations agreed with those obtained for the extractive solution analysis. The flavonoid content was expressed as grams of apigenin in 100 g of sample (Eq. 1). Each value represents the mean value of three determinations.

Scanning Electron Microscopy

The photomicrographs were taken with a Phillips XL20 (mod. PW 6620/00) scanning electron microscope (SEM).

RESULTS AND DISCUSSION

The extractive solution dried residue was 2.47 g%. The total flavonoid content of the extractive solution obtained by turbo-extraction was 0.082%. The spray-dried powders SDP1, SDP2, and SDP3 showed a residual moisture content of 7.58%, 9.17%, and 7.64% (w/w), respectively, so that the SDP2 and SDP3 moisture contents seem to be correlated with the amount of Gelita-Sol-P used.

The SPD particle size indicated the smallest diameter was that of the SDP3 regardless of the analysis method applied (Table 2, Fig. 1). For samples of the

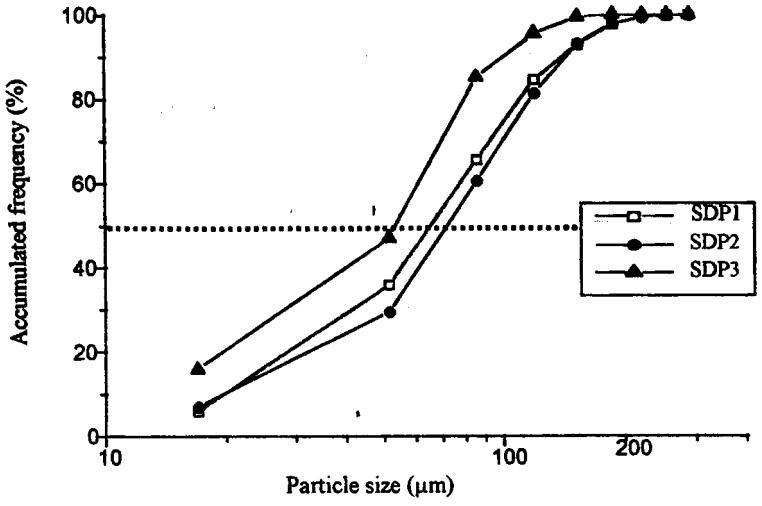

Figure 1. Graphic representation of spray-dried extract particle size distribution.

same dried powder, however, the results differed according to the calculation method, disregarding the fact that all spray-dried products presented a spherical form (Fig. 2). This was attributed to the asymmetrical curve distribution, which is corroborated by the kurtosis and skewness parameter determined for SDP1, SDP2, and SDP3 (Table 2). Since the RRSB log-cumulative curve was developed specifically for the statistical analysis of nonnormal distributions, the 85-, 85-, and 70-μm diameter values seemed to be more accurate theoretically. The particle size distribution curves were positively skewed (smaller size than the theoretical mean size) in a similar way as observed for milled products (14).

The SDP3 distribution curve amplitude (1.63) was narrower if compared with those of SDP1 (1.75) and SDP2 (1.70). These results agreed with the SDP1, SDP2, and SDP3 kurtosis values of −0.06, −0.91, and −0.90, respectively, as well as with the specific surface values of 138.2, 143.5, and 182.0 cm^2/cm^3, respectively. However, the specific surface values were not related to the particle mean diameter or to the surface-volume diameter (Table 2).

The SEM photographs showed for SDP1, SDP2, and SDP3 hollow, rough, and porous spherical particles (Fig. 2). The SDP2 and SDP3 showed, however, particles with a more irregular form together with a higher agglomeration tendency and poor flow properties.

Figures 3, 4, and 5 show the moisture uptake isotherms at 95%, 65%, and 35% RH atmospheres. At 95% RH, the moisture uptake increased remarkably during the first 2 days. At this time, the respective mass increases were 40.7% (w/w) for SDP1, 43.8% for SDP2, and 45.0% for SDP3. At the 14th day, the recorded values were 87.0% (w/w), 97.1% (w/w), and 92.3% (w/w), respectively, but the samples did not reach the equilibrium phase. The analysis of variance (ANOVA) test revealed significant differences between the last-mentioned values ($P = .05$). The SDP2 free-flow properties disappeared 24 hr after the experiment began. A similar occurrence was observed for SDP1 and SDP3, but 48 hr later. Four days later, all the samples became a more compact, harder, and darker color mass.

At 65% RH, the SDP1 showed a 7.0% (w/w) mass increase after 24 hr, while a 7.8% increase was observed for SDP2 and an 8.7% for SDP3. The equilibrium phase was achieved at the 6th day in all cases. The ANOVA test performed on the last day revealed significant differences among the moisture uptake of the powders ($P = .05$).

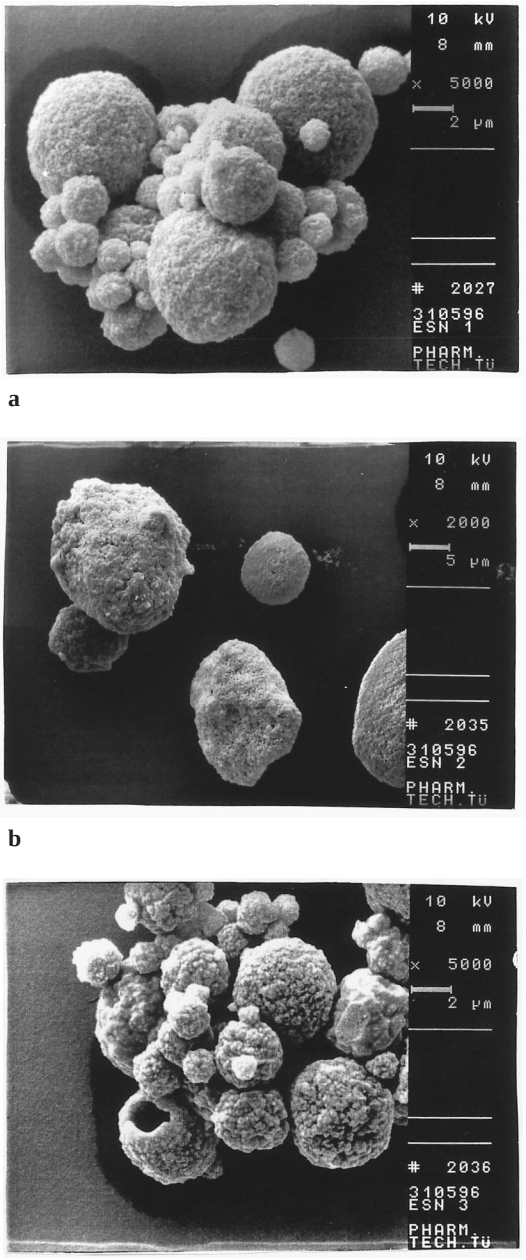

Figure 2. Scanning electron photomicrographs: (a) spray-dried powder with Aerosil 200 (SDP1); (b) spray-dried powder with Aerosil 200 (20 g) and Gelita-Sol-P (20 g) (SDP2); (c) spray-dried powder with Aerosil 200 (30 g) and Gelita-Sol-P (10 g) (SDP3).

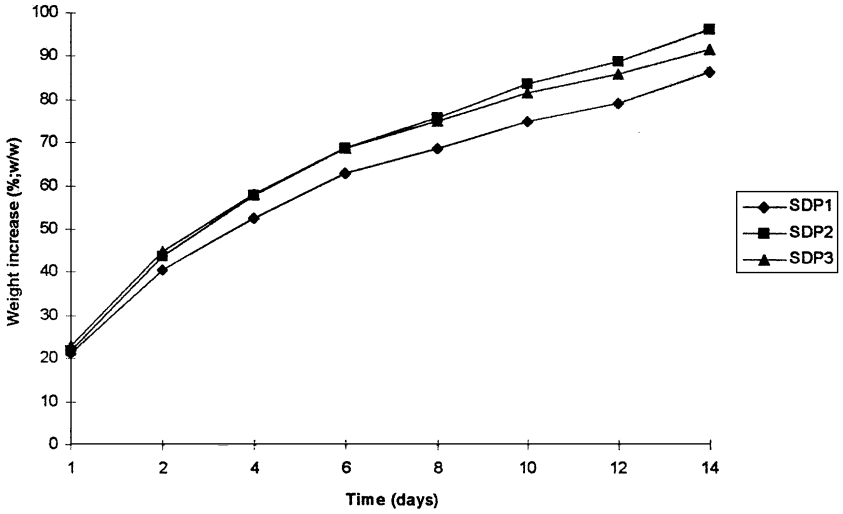

Figure 3. *Passiflora edulis* spray-dried powder moisture uptake isotherms at 95% RH.

At 35% RH, all the extracts showed a negligible mass loss on the first day, namely, 0.3% for SDP1, 1.4% for SDP2, and 1.6% for SDP3. From the first to the 14th day, little and not significant mass variations were observed.

The flavonoid content values obtained for SDP1, SDP2, and SDP3 were 2.55, 2.72, and 2.57 g%, respectively. Statistically, the flavonoid process recovery was no different (Student t test, $P = .05$).

CONCLUSIONS

The hygroscopicity and agglomerate tendency observed for the spray-dried products seem to be associated with the Gelita-Sol-P concentration. All spray-dried extracts were stable at 35% RH. All spray-dried extracts showed a nonnormal particle size distribution, so that the RRSB log-cumulative curve seems to be the most appropriate statistical method for the description of results.

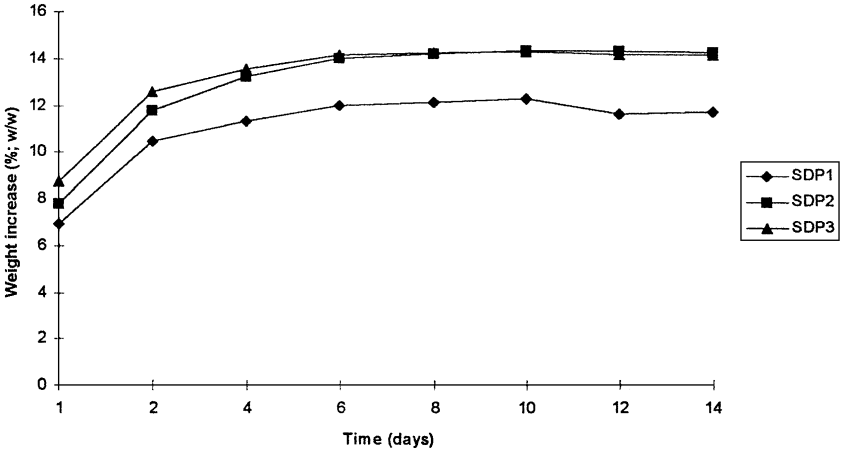

Figure 4. *Passiflora edulis* spray-dried powder moisture uptake isotherms at 65% RH.

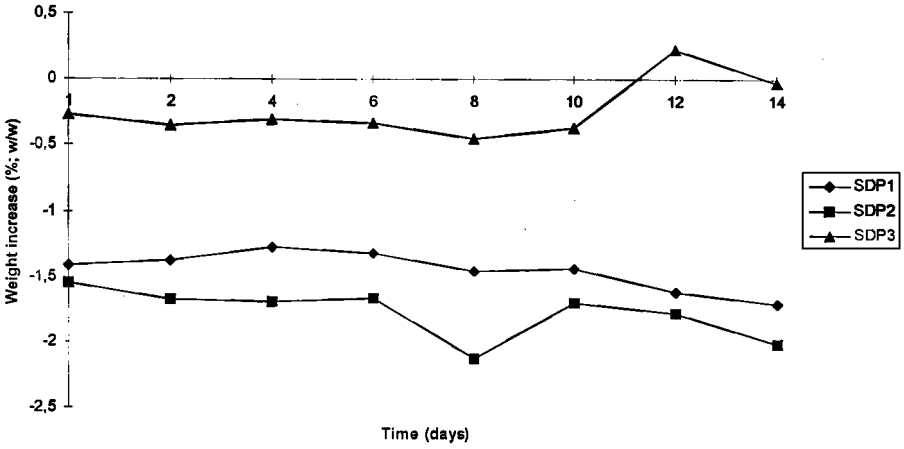

Figure 5. *Passiflora edulis* spray-dried powder moisture uptake isotherms at 35% RH.

The spray-dried powder results for the relative humidity behavior and total flavonoids contents analysis indicated better technological properties for the Aerosil 200 than achieved for Gelita-Sol-P.

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