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EVALUATION OF PHYSICO-CHEMICAL PROPERTIES OF SPRAY DRIED PINEAPPLE (*Ananas comosus* Merr.) POWDER PRODUCED WITH GUM ARABIC AS CARRIER AGENT

Le Pham Tan Quoc*

Institute of Biotechnology and Food Technology, Industrial University of Ho Chi Minh city, Ho Chi Minh city, Vietnam

*Corresponding Author: lephamtanquoc@iuh.edu.vn

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Abstract

The pineapple juice contains many bioactive compounds and they are quite sensitive to heat, light and oxygen. There are many methods to maintain these compounds, especially spray drying technology with gum arabic (GA) as a carrier agent. However, the physico-chemical properties of raw material and products should be determined to enhance storage stability. The received result showed that the pineapple juice was spray dried with 16% gum arabic (w/w) at drying air temperature of 160°C, output temperature of 70°C, airflow rate of 70 m³/h, feed flow rate of 750 mL/h and pressure of 4 bar. In addition, the current study also evaluated the changes in physico-chemical properties of gum arabic before and after the spray drying process including encapsulation yield, total polyphenol content (TPC), antioxidant activity (AA), moisture, bulk density, flowability, wettability, hygroscopicity, water solubility index (WSI), color parameters, structure and distribution of particles. In conclusion, GA has a significant influence on physico-chemical properties of powder produced by spray drying method. While the values of moisture, bulk density, wettability and a^* of powder product are lower than those of initial material, the opposite is true for the values of TPC, AA, hygroscopicity and WSI. It is noticeable that the values of flowability, L^* and b^* are relatively equally represented in both the initial material and powder product. In addition, the product has many various small sizes and its structure was smooth and spherical.

INTRODUCTION

Pineapple (*Ananas comosus* (L.) Merr.), is the only species in the Bromeliaceae family and a tropical plant with edible fruit. This plant is widely cultivated in the world, especially tropics and subtropics, for example, Asia (Thailand, Philippines, Indonesia, India and China), South Central America (Costa Rica and Brazil) and Africa (Nigeria and South Africa) [1]. In Vietnam, pineapple is considered as “the Queen of fruit” because of its rich nutrition, good taste, etc. It is commonly cultivated in Vietnam such as Nam Dinh, Thanh Hoa, Phu Tho province, especially Mekong delta (Kien Giang, Can Tho, Bac Lieu, Soc Trang province, etc.). Although there are many pineapple groups, only few groups are cultivated in Vietnam, for example, Cayenne, Queen and

Spanish pineapple. They were cholesterol-free or fat-free, low in sodium or calories and have high nutrients (fiber, antioxidants, bromelain, manganese, copper, vitamin C, vitamin B complex, calcium, zinc and β -carotene) [1, 2]. Therefore, nowadays, pineapple is applied in jam or juice industry in Vietnam. The high content of vitamin C in fruit is quite useful for human health to prevent some diseases such as common cold and joint pain. Besides, antioxidants in this fruit play an important role in antioxidative stress, prevent cancers and reduce inflammation, etc. [3-5].

In fact, pineapple has many benefits to human health due to the bioactive compounds and is widely applied in food industries such as canned pineapple, candied pineapple, cake, juice, dehydrofrozen pineapple, pineapple concentrate,

yogurt fortified with pineapple peel powder, pineapple leather, etc. [6]. However, these compounds easily degrade in the surrounding atmosphere and it is quite difficult to preserve. Hence, the encapsulation of pineapple extract using spray drying is the optimal method applied for the production, stabilization of bioactive compounds and extension of shelf-life of the product. In there, using gum arabic (GA) as a carrier agent is the best choice.

To date there are not any studies that only used GA to spray-dry the pineapple juice. Hence, this study was carried out to evaluate the physico-chemical properties of pineapple fruit powder, with emphasis on TPC, AC, recovery efficiency, wettability, water solubility index (WSI), bulk density, flowability, hygroscopicity, the particle size and shape of microencapsulation agent.

MATERIALS AND METHODS

Chemicals and Reagents

Gum arabic (GA) was supplied by Xiamen Ditai Chemicals company (China). Folin Ciocalteu, DPPH (2,2-diphenyl-1-picrylhydrazyl) and Trolox reagents (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic) were bought from Sigma-Aldrich (USA). Besides, other chemicals used were of analytical grade.

Sample Preparation

The pineapples were washed, peeled, removed eyes and cut into small pieces with a size of 3×3 cm. Then, the pineapple flesh was crushed in a Philips blender for 30 seconds. The mixture was added 1.5% pectinase (v/w) (Pectinex Ultra SP-L) and incubated in the thermostatic baths at 50°C for 1.5 hours. After that, it was pressed to squeeze out the juice. The total soluble solids (TSS) of the received extract was adjusted to 12°Brix and it is preserved at 4°C. In this study, GA was added to the pineapple juice with the concentration of 16% (w/w). This mixture was filtered, and spray dried by LabPlant SD-Basic Spray Dryer (England) at the inlet/outlet air temperature of 160°C/70°C, 4 bar, airflow rate of 70 m³/h and feed flow rate of 750 mL/h.

Encapsulation Yield

According to Roccia et al., EY was calculated as the ratio of the powder weight collected after every spray drying experiment to the initial amount of solids in the sprayed dispersion volume [7].

$$H = \frac{m_1}{m_2} \times 100 (\%)$$

where m_1 : The total soluble solids of the spray dried powders (g) m_2 : The total soluble solids of the feed weight (g)

Total Polyphenol Content (TPC)

TPC was determined based on the Folin Ciocalteu method with some slight modifications [8]. The results were based on a standard curve obtained with gallic acid. TPC was expressed as mg of gallic acid equivalent per gram of dry weight (mg GAE/g DW).

Antioxidant Activity (AA)

The AA of material and product was determined by DPPH assay with some slight modifications [9]. Trolox was used as the standard. AA was expressed as μ mol of Trolox equivalent per gram of dry weight (μ mol TE/g DW).

Bulk Density

Powder (2 g) was added into an empty graduated cylinder (10 mL) and shook for 1 min. The result was determined by the ratio of mass of the powder and the final volume occupied by the powder in the cylinder [10].

Flowability

According to Geldart et al., flowability was determined by the measurement of the angle of repose (AOR) [11] with some slight modifications. The funnel was held at a fixed height on the flat base (5 cm) and the powder (15 g) was slowly poured through the funnel to form to a cone. Then, the inverse tangent of the ratio of height and half of width (radius) of the base of the cone was the AOR.

Wettability

This parameter was determined by the method of Freudig et al. [12] with slight modifications. The powders were poured into the funnel which was held at a fixed height and fell down a volume of 100 mL water in a beaker (250 mL) at room temperature. After that, the time for the whole amount of powder to visibly sink beneath the water surface was recorded as an indicator of wettability.

Hygroscopicity

About 1.5 g samples were placed in an airtight plastic container at 25°C containing the saturated solution of sodium carbonate. These samples were weighed after 7 days. Eventually, hygroscopicity was determined as gram of adsorbed moisture per 100 g solids (g/100 g) [13].

Water Solubility Index (WSI)

The method to determine WSI was adapted from Anderson et al. [14] with some small changes. Powder (1 g) and deionized water (10 mL) were vigorously mixed for 1 min. Then, the mixture was made up to 15 mL by deionized water, moved to the centrifuge tube and incubated for 30 minutes at 37°C. After that, it was centrifuged for 30 minutes at 8000 rpm; the supernatant was separated and dried at 103°C in an oven. The WSI (%) was expressed as the percentage of dried supernatant to the amount of original powder.

Color Parameters

The color parameters consist of L^* (lightness), a^* (redness and greenness) and b^* (yellowness and blueness) values, which were determined by a Chroma Meter CR-400 (Minolta, Japan).

Scanning Electron Microscopy (SEM)

The morphology of the initial material and the spray dried powder was examined by a Jeol/JSM-7401F scanning electron microscope system (Japan). All samples were put on SEM stubs using a double-sided adhesive tape and coated a thin layer of gold/platinum in vacuum condition. Then, they were observed at a magnification of 1000 \times .

Particle Size And Distribution Analysis

A laser scattering particle size distribution analyzer (HORIBA LA-960, Japan) was used to determine the particle sizes of material and spray dried powder.

Statistical Analysis

The experimental data was analyzed by the one-way analysis of variance (ANOVA) method and significant differences among the means from triplicate analysis at $p < 0.05$ were determined by Fisher's least significant difference (LSD) procedure using Statgraphics software (Centurion XV). The values obtained were expressed in the form of a mean \pm standard deviation (SD).

Table 1. EY, moisture, TPC and AA of carrier agents and powder products

Sample	EY(%)	Moisture (%)	TPC (mg GAE/g DW)	AA (μ mol TE/g DW)
GA	-	11.37 \pm 0.22 ^a	0.87 \pm 0.02 ^a	1.64 \pm 0.20 ^a
GA _p	39.59 \pm 2.67	3.52 \pm 0.22 ^b	2.57 \pm 0.02 ^b	6.28 \pm 0.21 ^b

Different letters in the same column indicate a statistically significant difference ($p < 0.05$) between the initial material and product. GA_p: gum arabic and pineapple juice after spray drying process.

RESULTS AND DISCUSSION

EY, Moisture, TPC and AA of Powder

The results in Table 1 show that the recovery efficiency of GA is 39.59%, it was lower than that in study of Quoc and Muoi, who added GA into the *P. multiflorum* Thunb. root extract reaching 15% soluble solids, EY reached 65.17% [15]. In addition, some studies noticed that EY from using only GA was lower than other carrier agents because GA had a short-chain branched structure, high hydrophilic nature [16, 17]. Therefore, the properties of this material easily assisted the adhesion of particles on the spray dryer wall.

The moisture of GA_p (3.52%) was lower than that of initial GA (11.37%) and that in study of Do and Nguyen (3.9-5.15%). They spray dried mulberry extract with various inlet temperatures and ratios of GA to microcrystalline cellulose [18]. In general, the moisture of this product was less than 5%, which was quite safe to store, limiting spoilage due to low water activity. Differences in moisture can be explained by the differences in materials, spray drying conditions such as airflow rate, inlet temperature, etc. [10].

Results show that there was an increase in TPC and AC of powder before and after spray drying. To be specific, TPC increased sharply from 0.87 to 2.57 mg GAE/g DW, AA increased dramatically from 1.64 to 6.28 μ mol TE/g DW. This shows that this spray drying method was capable of keeping the precious biological compounds found in fruits or other herbs. According to Sensoy et al., the spray drying involves the use of heat, therefore it affects the antioxidant ability [19]. TPC of GA_p was lower than that of spray dried products from *P. multiflorum* Thunb. root extract [15] and mulberry juice [18]. However, it was higher than that of spray dried powder from watermelon and pineapple juice [20]. Besides, AA of GA_p also was quite low compared with that of other materials such as combination of maltodextrin and GA [15]; combination of maltodextrin, GA and microcrystalline cellulose [18]. Materials and spray drying temperatures are the main causes of these differences.

Bulk Density, Flowability, Wettability and Hygroscopicity of Powder

The results show that the bulk density of GA decreased sharply after spray drying process (from 0.84 g/mL to 0.65 g/mL) (Table 2). Bulk density of GA_p was higher than that of spray dried GA powder from *P. multiflorum* Thunb. root extract (0.53 g/mL) [15] and spray dried MD powder from four juices [20]. The decrease in bulk density can be

explained as follow: when dried at high temperatures, the formation of particles occurs quickly, the surface of the drops of liquid dries quickly and forms a waterproof layer, then forming bubbles vapor and droplets increased in size leading to a reduction of density [21]. In addition, other studies also suggested that the bulk density of spray dried products are affected by various factors such as speed feed stream, air flow rate, injection pressure, etc. [22].

Table 2. Bulk density, flowability, wettability and hygroscopicity of powder products

Sample	Bulk density (g/mL)	Flowability (AOR, °)	Wettability (s)	Hygroscopicity (g/100 g)
GA	0.84 ± 0.02 ^a	34.26 ± 0.62 ^a	4692.67 ± 410.67 ^a	6.3 ± 0.57 ^a
GA _p	0.65 ± 0.02 ^b	34.23 ± 1.03 ^a	701.67 ± 10.21 ^b	21.21 ± 1.74 ^b

Different letters in the same column indicate a statistically significant difference ($p < 0.05$) between the initial material and product. GA_p: gum arabic and pineapple juice after spray drying process.

Flowability was evaluated through an angle of repose (AOR). The difference of AOR of GA before and after the spray drying process is negligible. This result is not similar to other studies. In particular, GA powder from *P. multiflorum* Thunb. root extract after spray drying increased steadily from 31.13° to 37.66°. However, this result is in agreement with the statement of Carr, AOR of GA ranges from 30° to 40° [23]. The flow of powder may also be significantly affected by the relative humidity of ambient air. The desiccant tends to dissolve the compounds on the surface, forming links bridge between particles making them more cohesive [24]. Besides, particle size is a major factor that strongly affects the flowability of the powder. When the particle size is reduced, surface area per unit mass of powder rises and this leads to the changes of flowability [25]. Essentially, there are many causes leading to changes in flowability, especially carrier agents.

Wettability of GA tends to decrease compared with the product after the spray drying process. It reduces from 4692.67 to 701.67 sec. This result is contrary to the study of Quoc and Muoi, in which the wettability of GA increased after spray drying [15]. In addition, the wettability of GA_p is higher than that of blackberry GA powder (134.2 sec) [26]. The previous study also pointed out that the shape, particle size and carrier agents strongly affected wettability, especially moisture of powder. Water easily penetrated into powder if the moisture of powder is low [27].

After the spray drying process, hygroscopicity increased significantly from 6.3 to 21.21 g/100 g. The difference of hygroscopicity depends on the type of carrier agents and initial material, for instance, spray dried GA powder of *P. multiflorum* Thunb. root extract and beetroot juice was 37.16 g/100 g and 18.11 g/100 g, respectively [15, 17], while spray dried MD powder of *Amaranthus* extract was from 44.6 to 49.5 g/100 g. Besides, hygroscopicity could be affected by the moisture of material and spray drying temperature. High hygroscopicity can be seen as a disadvantage while preserving the bioactive compositions inside the core [13].

Water Solubility Index (WSI) and Color Parameters of Powder

WSI of powder product increased dramatically from 70.78 to 88.23% after spray drying process (Table 3). In general, WSI of spray dried powder tends to be higher than that of original powder. In addition, this result shows that WSI of GA is lower than that of other carrier agents, for instance, WSI of spray dried MD powder of *P. multiflorum* Thunb. extract and amla juice were 94.5% and 91.43%-94.98%, respectively [15, 28]. This also proves that WSI depends on carrier agents and the initial extract. Besides, some previous studies pointed out that the concentration of carrier agents, spray drying temperature and air flow rate also strongly affect WSI [29, 30].

Table 3. Water solubility index (WSI) and color parameters of powder products

Sample	WSI (%)	<i>L</i> [*]	<i>a</i> [*]	<i>b</i> [*]
GA	70.78 ± 2.96 ^a	87.98 ± 0.44 ^a	0.99 ± 0.12 ^a	16.27 ± 0.47 ^a
GA _p	88.23 ± 2.71 ^b	85.23 ± 2.3 ^a	0.04 ± 0.06 ^b	15.49 ± 0.79 ^a

Different letters in the same column indicate a statistically significant difference ($p < 0.05$) between the initial material and product. GA_p: gum arabic and pineapple juice after spray drying process.

The color parameters after spray drying change insignificantly including L^* and b^* value, while a^* value decreased slightly from 0.99 to 0.04. Changes of color are due to Maillard reaction and caramelization that occur during the spray drying process. The main causes are the presence of high sugar content in the juice and high temperature in spray drying chamber.

Microstructure and Particle Size of Spray Dried Powder

Based on the received results, electron micrographs recorded the successful formation of microcapsules for GA_p . GA particles were found to be in irregular shapes while GA_p particle was smooth and spherical (Figure 1). The surface of GA_p particles has few small indentions and wrinkles. No traces of irregular shape particles were found for spray dried products, this proves that there is a possible conversion of GA from an amorphous form into a composite particle. The similar microstructure was observed in microcapsules of *P. multiflorum* Thunb. extract with GA [15], betalain extract with maltodextrin and cladode mucilage [31], etc. The changes in microstructure of spray dried products depend on carrier agents and inlet air temperature [30].

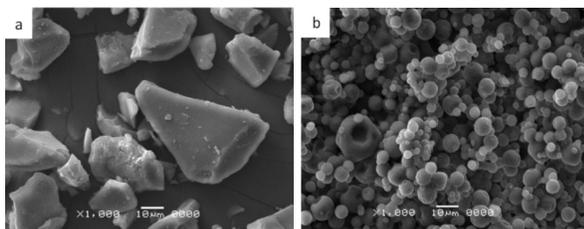


Figure 1. The external morphology of carrier agent (a) and spray dried product (b)

The particle size of GA_p changed dramatically and was smaller than the initial material. The diameter of GA ranges from 3.9 to 451.5 μm ($d_{\text{mean}}=60.16 \mu\text{m}$), while GA_p was from 4.47 to 451.5 μm ($d_{\text{mean}}=27.53 \mu\text{m}$) (Figure 2). Especially, GA_p has many sizes compared with GA and this result is similar to that in the study of Quoc and Muoi on *Polygonum multiflorum* Thunb. powder with GA , maltodextrin as carrier agents [15]. However, the average diameter of spray dried product differs from that of other studies, for instance, blackberry GA powder ($d_{\text{mean}}=10.98 \mu\text{m}$) [26], *M. citrifolia* L. κ -carrageenan powder ($d_{\text{mean}}=2.27 \mu\text{m}$), *M. citrifolia* L. maltodextrin powder ($d_{\text{mean}}=4.82 \mu\text{m}$) [32]. The particle size of product depends on carrier agents, inlet temperature, flow rate [13, 21, 33], injection pressure [22], etc. Almost properties of spray dried powder (bulk density, wettability, hygroscopicity, AOR, WSI, etc.) depends on the particle size.

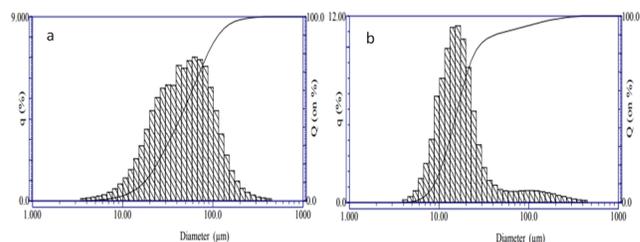


Figure 2. Particle size distribution of carrier agent (a) and spray dried product (b)

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CONFLICT OF INTEREST

The author declares no conflict of interests regarding the publication of this manuscript.

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