

Technical Parameters Affecting the Spray Drying of Roselle (*Hibiscus Sabdariffa*) Powder

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Abstract

Roselle (*Hibiscus sabdariffa*) was a member of Malvaceae family. Its calyxes had bright red color due to presence of anthocyanin with an excellent antioxidant property. Raw roselle (*Hibiscus sabdariffa* L.) calyx was highly perishable due to its high moisture content. In order to diversify products from this plant, this research evaluated the possibility of spray drying for roselle extract into dried powder for long-term consumption. We focused on the effect of sugar alcohols (mannitol, sorbitol, isomalt, xylitol, erythritol) at 8%, carrier agents (maltodextrin, gum arabic, glutinous starch, whey protein concentrate, carboxymethyl cellulose) at 12%, operating parameters of spray dryer (inlet/outlet air temperature, feed rate) on physicochemical quality (bulk density, solubility, total phenolic content, total flavonoid content, anthocyanin content) of roselle powder. Results showed that the optimal spray drying variables for roselle powder should be 8% isomalt, 12% whey protein concentrate, inlet/ outlet air temperature 140/85°C/°C, feed rate 12 ml/min. Based on these optimal conditions, the highest physicochemical attributes of the dried roselle calyx powder would be obtained.

Keywords: Anthocyanin, carrier agent, operating parameter, physicochemical, Roselle calyx, sugar alcohol

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INTRODUCTION

Spray drying was widely utilized in food industry to preserve thermal-sensitive phytochemical constituents in herbs, vegetables and fruits in which solvent mixture was sprayed and transformed into crystallize substances with appropriate attributes¹⁻¹⁰. The degree of crystallization was accelerated when the moisture content was reduced and when the concentration of solid material was increased^{3,11,12}. Spray dried powder was convenient for handling and distribution by its light weight¹³. Water mobility in the food matrix caused changes of physicochemical, microbial and nutritional properties of spray dried powder^{13,14}. Moisture was commonly absorbed into spray dried powder under preservation. Spray drying would become an ideal approach if the shelf life of the powder was extended and the antioxidant elements in material extract could be preserved during production, and if the contacting surface area could be extended¹⁵.

Roselle (*Hibiscus sabdariffa* L.) calyx drew a special attention due to its nutritional composition, bioactive constituents and therapeutic applications¹⁶. Extract from roselle calyces had unique pigment by the existing of anthocyanin with high antioxidant properties^{17,18}. It's beneficial to relieve hypertension, hyperlipidemia, cancer and inflammatory diseases¹⁹. It's also demonstrated to mild the blood viscosity, stimulate milk release and induce intestinal peristalsis²⁰⁻²². Roselle (*Hibiscus sabdariffa* L.) calyx commonly utilized to convert into value added products like wine, syrup, juice, jam, jelly, pudding, cake, ice cream, tea, confectionary, sauce, marmalade, chocolate, flavouring agent^{16,23-31}. There were several notable reports mentioned to the processing of roselle calyx. The influences of input air temperature and feed rate on the powder quality attributes during spray-drying of roselle extract were examined²⁹. Various variables in production of dried Roselle (*Hibiscus sabdariffa* L.) calyx herbal tea were investigated¹⁶. Archaina et al.³² maintained the antioxidant ability and enhanced the shelf life of spray-dried roselle powder by incorporation of maltodextrin and gum arabic as wall material. Al-Kahtani HA and Hassan BH³³ manufactured spray-dried powder from extract of roselle (*Hibiscus sabdariffa*) calyces. Gonzalez-Palomares et al.³⁴ verified different drying temperatures

on the volatile substances and organoleptic property of the roselle (*Hibiscus sabdariffa*) powder. Mohamad et al.³⁵ examined the the influence of air temperature of foam mat drying on the kinetics and quality of dehydrated roselle extract. Cid-Ortega and Guerrero-Beltran³⁶ verified the microencapsulation of *Hibiscus sabdariffa* (roselle) extract by spray drying with the support of maltodextrin and gum arabic as wall material. Navidad-Murrieta et al.³⁷ demonstrated the effect of maltodextrin and gum arabic as carrier material in spray-drying of *Hibiscus sabdariffa* extract. Objective of our study to examine the effect of sugar alcohols (mannitol, sorbitol, isomalt, xylitol, erythritol), carrier agents (maltodextrin, gum arabic, glutinous starch, whey protein concentrate, carboxymethyl cellulose), operating parameters of spray dryer (inlet/outlet air temperature, feed rate) on physicochemical quality (bulk density, solubility, total phenolic content, total flavonoid content, anthocyanin content) of rosselle powder.

MATERIAL AND METHOD

Material

Hibiscus sabdariffa calyces were collected in Soc Trang province, Vietnam. Fully bloomed, disease free, and undamaged healthy calyces were chosen. They were washed under clean water to remove foreign matters. Chemical reagents such as FolinCiocalteu's reagent, NaHCO₃, gallic acid, AlCl₃, methanol, quercetin were all analytical grade purchased in Ho Chi Minh city, Vietnam. Powder was obtained by using a mini spray dryer.

Researching method

Hibiscus sabdariffa calyces were primarily frozen-thawed, twice pressed and extracted by hot water in ratio 1: 4 (material: water, w/v) at 90°C for 15 minutes. The mixture was passed through vacuum filter to collect extract.

Experiment #1

The *Hibiscus sabdariffa* extract was added with 8% sugar alcohols (mannitol, sorbitol, isomalt, xylitol, erythritol) before undergoing spray drying process by 12% maltodextrin as carrier agent, inlet temperature 130°C, outlet temperature 75°C, feed rate 8 ml/min.

Experiment #2

The *Hibiscus sabdariffa* extract was added 8% isomalt plus 12% carrier agents (gum arabic, maltodextrin, modified starch, whey

protein concentrate, carboxy methylcellulose). This mixture was ready for spray drying at inlet temperature 130°C, outlet temperature 75°C, feed rate 8 ml/min.

Experiment #3

The *Hibiscus sabdariffa* extract was added 8% isomalt plus 12% whey protein concentrate. The spray dryer conducted at different inlet/outlet air temperature (130/75, 135/80, 140/85, 145/90, 150/95, °C/°C) at feed rate 8 ml/min. After choosing the appropriate inlet/outlet air temperature, the spray drying process also verified the effect of different feed rate (8, 10, 12, 14, 16 ml/min).

The target functions in each experiment were based on bulk density (mg/ml), solubility (%), total phenolics (mg GAE/ 100 g), total flavonoids (mg QE/ 100 g), anthocyanin (mg/ 100 g) of the spray-dried powder.

Physicochemical evaluation

Bulk density (g/ml) and the solubility (%) were determined by procedures described by Saikia et al.⁷ A 5 g of dehydrated sample was poured into a 50 mL graduated cylinder, and measured the line fullfilled by the powder as bulk density. 5 g powder was dissolved with distilled water at a ratio of 1:9. The fluid was then separated by centrifugator at 3,500 rpm within 4 minutes. The supernatant was obtained, dehydrated and measured the weight. The solubility (%) was estimated by dividing the weight of supernatant after dehydration by the weight of primary powder, multiplying with 100. Total phenolic (mg GAE/100g) content was determined using spectrophotometric method³⁸. 5 ml of methanol was mixed with 5 g powder,

25ml of 9.5% FolinCiocalteu's reagent dissolved in distilled water and 25 ml of 7.0% NaHCO₃. The control sample was diluted, including 5ml methanol, 25ml of 9.5% Folin-Ciocalteu's reagent dissolved in distilled water and 25ml of 7.0% of NaHCO₃. Samples were then incubated at 50°C for 20 minutes. The absorbance was recorded at λ_{max} =765nm by a spectrophotometer. The similar protocol was conducted for the standard solution of gallic acid and the calibration line was established. Read on the absorbance value, the total phenolic concentration was calculated from the calibration curve; the total phenolic concentration was presented as a gallic acid equivalence (mg of GAE/100g of sample). Total of flavonoids (mg QE/100g) was examined using spectrophotometric technique³⁹. 25 gram of powder was extracted with 5 ml of methanol with the presence of 5 ml of 2.5% AlCl₃ solution. The samples were kept for 1.5 hours at ambient condition. This mixture was incubated for 25 minutes at ambient condition. The absorbance was verified at λ_{max} =415nm by a spectrophotometer. The similar protocol was prepared for the standard solution of quercetin and the calibration graph was established. Read on the calculated absorbance, the flavonoid concentration was calculated (mg/mL) from the calibration curve and the flavonoid concentration was presented as a term of quercetin equivalent (mg of QE/100g of sample). Anthocyanin content (mg/100g) was determined by spectroscopy at wavelength 530 nm⁴⁰.

Statistical summary

The demonstrations were prepared in 3 times for various sample groups. The values were

Table 1. Effect of sugar alcohols (8%) on physicochemical property of *Hibiscus sabdariffa* powder

Sugar alcohols	Mannitol	Sorbitol	Isomalt	Xylitol	Erythritol
Bulk density (g/ml)	0.29±0.01 ^c	0.36±0.00 ^{bc}	0.69±0.03 ^a	0.57±0.01 ^{ab}	0.48±0.02 ^b
Solubility (%)	48.75±0.03 ^c	52.68±0.02 ^{bc}	77.42±0.01 ^a	69.83±0.00 ^{ab}	60.51±0.03 ^b
Total phenolic (mg GAE/ 100 g)	297.15±1.17 ^c	328.64±1.63 ^{bc}	487.50±1.52 ^a	401.27±1.19 ^{ab}	359.34±1.32 ^b
Total flavonoid (mg QE/ 100 g)	116.21±0.34 ^c	154.36±0.18 ^{bc}	224.67±0.23 ^a	201.42±0.17 ^{ab}	186.04±0.22 ^b
Anthocyanin (mg/100g)	79.83±0.11 ^c	93.68±0.07 ^{bc}	185.16±0.15 ^a	145.63±0.20 ^{ab}	119.71±0.07 ^b

Figures are the mean of three replications; Figures in row followed by the same letter/s are not differed significantly ($\alpha = P=0.05$).

expressed as mean±standard deviation. Statistical summary was conducted by the software of Statgraphics XVI.

RESULT AND DISCUSSION

Effect of sugar alcohols on physicochemical property of *Hibiscus sabdariffa* powder

Table 1 presented the influence of different sugar alcohols (mannitol, sorbitol, isomalt, xylitol, erythritol) supplemented on physicochemical attributes of roselle calyx powder. Isomalt revealed the best candidate to replace saccharose as well as other sugar alcohols. Mannitol, the sugar alcohol stereoisomer of sorbitol, originated from starch or sugar by hydrogenation, by fermentation or by natural product extraction⁴¹⁻⁴². Mannitol, mildly acidic in aqueous solution, exhibited sweetness equal to glucose and half as sweet as sucrose. Mannitol played as an osmotic diuretic agent and weak renal vasodilator⁴³. Sorbitol had good compressibility and commonly utilized as a bulking agent⁴⁴. Isomalt originated from sugar by fermentation of sucrose to isomaltulose and further hydrogenation of the reducing fructose moiety. Isomalt exhibited about half the sweetness of sucrose. Xylitol was prepared from hydrolyzation of xylan into xylose which was catalytically hydrogenated into xylitol. Xylitol had sweetness as sucrose and gave an excellent cooling effect. Erythritol was a 4-carbon sugar alcohol obtained by yeast fermentation of glucose or sucrose. Its sweetness was 80% relative to sucrose combined with the strongest cooling effect.

Bulk density was the weight of the solid components with water divided by the total volume

fulfilled by the particles, layer moisture and all pores inside⁴⁵. The advantage of powder obtained with high bulk density was that it could be kept in high amount into small container and vice versa⁴⁵. At low moisture content, the spray dried powder was very easy to absorb water in surrounding atmosphere to get sticky and inconvenient to manipulate⁴⁵. At low bulk density, the high amount of air existed within the powder and more chance of powder oxidation and shorter preserved shelf life⁴⁶. Low bulk density indicated in high space for packaging. Accelerating in bulk density was greatly related to moisture content absorbed and water activity built up⁴⁷⁻⁴⁸. The soluble capacity of powder was an important quality property of spray dried powders⁴⁹. Solubility reflected the final quality of powder that could be brought into stable suspension⁵⁰. Solubility was related to simplicity of reconstitution. Poor solubility happened especially at high temperature and high solid content. Functional solubility was highly affected by structural modification of the powder⁵¹. High concentration of drying agents induced to low solubility of the dried fruit powder⁵². The higher the solubility of powder, the lower the bulk density⁵³. Solubility was one of major problems in the dried powder obtained from spray drying due to existing of low molecular weight sugars and acids⁵⁴. Gum resulted in high solubility in aqueous solution facilitating the spray drying⁵⁵. Anthocyanins were hydrophilic pigment specifically compatible with a water-based gel. Phenolics and flavonoids might create complexes with polysaccharides and the affinity of phenolics to polysaccharides depended on the water solubility, molecular

Table 2. Effect of carrier agents (12 %) on physicochemical property of *Hibiscus sabdariffa* powder

Carrier agents	Gum arabic	Maltodextrin	Modified starch	Whey protein concentrate	Carboxy methyl cellulose
Bulk density (g/ml)	0.50±0.01 ^{bc}	0.69±0.03 ^b	0.75±0.02 ^{ab}	0.84±0.01 ^a	0.39±0.02 ^c
Solubility (%)	69.34±0.00 ^{bc}	77.42±0.01 ^b	80.17±0.03 ^{ab}	85.49±0.00 ^a	60.08±0.03 ^c
Total phenolic (mg GAE/ 100 g)	443.18±1.45 ^{bc}	487.50±1.52 ^b	501.23±1.64 ^{ab}	520.67±1.26 ^a	401.24±1.32 ^c
Total flavonoid (mg QE/ 100 g)	198.50±0.18 ^{bc}	224.67±0.23 ^b	248.36±0.12 ^{ab}	271.52±0.17 ^a	160.43±0.21 ^c
Anthocyanin (mg/100g)	160.24±0.21 ^{bc}	185.16±0.15 ^b	201.13±0.09 ^{ab}	229.54±0.15 ^a	146.81±0.13 ^c

Figures are the mean of three replications; Figures in row followed by the same letter/s are not differed significantly ($\alpha = P=0.05$).

size, conformational mobility and structure of polyphenol. Anthocyanins and polyphenols were easy decomposed under thermal processing⁵⁶.

Influence of carrier agents on physicochemical property of *Hibiscus sabdariffa* powder

Wall material agents significantly influenced the anthocyanin stability of roselle calyx⁵⁷. Various carriers was used in spray drying of roselle to encapsulate anthocyanin such as pullulan⁵⁸, trehalose and maltodextrin⁵⁹, maltodextrin and gum Arabic⁶⁰. Table 2 showed the influence of different sugar carrier agents on physicochemical attributes of roselle calyx powder. Among gum arabic, maltodextrin, modified starch and carboxymethyl cellulose; whey protein concentrate revealed the best option for spray drying roselle calyx powder (bulk density 0.84±0.01 g/ml, solubility 85.49±0.00%, total phenolic 520.67±1.26 mg GAE/ 100 g, total flavonoid 271.52±0.17 mg QE/ 100 g, anthocyanin 229.54±0.15 mg/100g). Meanwhile, carboxymethyl cellulose resulted to the lowest physicochemical properties of roselle calyx powder (bulk density 0.39±0.02 g/ml, solubility 60.08±0.03%, total phenolic 401.24±1.32 mg GAE/ 100 g, total flavonoid 160.43±0.21 mg QE/ 100 g, anthocyanin 146.81±0.13 mg/100g). Yousefi et al.⁴⁵ examined maltodextrin, gum Arabic, waxy starch as carrier agent on on the physicochemical properties of pomegranate powder. Gum arabic revealed the most effective yield, bulk density and solubility. Cristhiane et al.⁶¹ examined the impact of maltodextrin, gum arabic on the physicochemical characteristics of blackberry powder produced by spray dehydration. Maltodextrin contributed to

less water absorption of powder with low water activity and high dissolving attributes. Bhusari et al.⁶² examined influence of wall materials on physical and microtextural characteristics of spray-dehydrated tamarind pulp powder. Whey protein concentrate revealed the best improvement of yield, color, bulk density and hygroscopicity. Tan and Thuy⁶³ optimized maltodextrin and arabic gum during spray drying of *Pouzolzia zeylanica* extract. The optimum concentrations of 8.74% maltodextrin and 0.08% arabic gum were concluded. Varastegani et al.⁶⁴ evaluated the influence of spray-drying with maltodextrin and gum arabic on antioxidant capability, ash, proximate and overall acceptance. Data revealed that spray-dehydrated powder had low caffeine and fat content, antioxidant activity. Meanwhile the ash of spray-dehydrated product strongly accelerated. Archaina et al.³² concluded that using maltodextrin-gum arabic as wall material resulted spray-dired roselle powder with excellent physical attributes such as low moisture content, low cohesiveness, high glass transition temperature, high solubility, good flowability. Cid-Ortega and Guerrero-Beltran³⁶ verified the microencapsulation of *Hibiscus sabdariffa* (roselle) extract by spray drying with the support of maltodextrin and gum arabic as wall material. The best formula of carrier agent was noticed at 3% maltodextrin: gum arabic. Navidad-Murrieta et al.³⁷ demonstrated the effect of maltodextrin and gum arabic as carrier material in spray-drying of *Hibiscus sabdariffa* extract. Results revealed that maltodextrin: gum arabic (80:20, w/w) was appropriate to encapsulate and retain the most phenolics in *Hibiscus*

Table 3. Effect of inlet/ outlet temperature (oC) on physicochemical property of *Hibiscus sabdariffa* powder

Inlet/ outlet temp. (°C/°C)	130/75	135/80	140/85	145/90	150/95
Bulk density (g/ml)	0.84±0.01b	0.89±0.03ab	0.93±0.00a	0.77±0.02bc	0.68±0.01c
Solubility (%)	85.49±0.00b	87.33±0.02ab	90.04±0.03a	80.25±0.00bc	74.30±0.03c
Total phenolic (mg GAE/ 100 g)	520.67±1.26a	507.30±1.13ab	484.12±1.37b	454.12±1.05bc	423.07±1.18c
Total flavonoid (mg QE/ 100 g)	271.52±0.17a	234.39±0.23ab	201.57±0.12b	180.03±0.14bc	161.25±0.09c
Anthocyanin (mg/100g)	229.54±0.15a	197.30±0.11ab	164.29±0.13b	120.74±0.08bc	99.63±0.07c

Figures are the mean of three replications; Figures in row followed by the same letter/s are not differed significantly ($\alpha = P=0.05$).

Impact of spray dehydration parameters (inlet/outlet temperature, feed rate) on physicochemical property of Hibiscus sabdariffa powder

Table 3 and 4 showed the influence of inlet/ outlet temperature, feed rate on physicochemical attributes of roselle calyx powder. Roselle calyx extract should be dehydrated at 140/85 (°C/°C) of inlet/ outlet temperature by 12 ml/min of feed rate to obtain the optimal bulk density 0.76±0.01 g/ml, solubility 78.25±0.02%, total phenolic 519.14±1.08 mg GAE/ 100 g, total flavonoid 268.65±0.10 mg QE/ 100 g, anthocyanin 230.07±0.13 mg/100g. Our present study was similar to other following findings: Al-Kahtani H. A. and Hassan B. H.³³ manufactured spray-dried powder from extract of roselle (*Hibiscus sabdariffa*) calyces. They confirmed that the optimal inlet air temperature was noticed at 198.5°C to obtain the best protein content, ascorbic acid, and solubility of the spray-dried powder. Gonzalez-Palomares et al.³⁴ verified different drying temperatures on the volatile substances and organoleptic property of the roselle (*Hibiscus sabdariffa*) powder. They concluded that the best powder should be conducted at temperature between 190°C and 200°C. Mohamad et al.³⁶ examined the the influence of air temperature of foam mat drying on the kinetics and quality of dehydrated roselle extract. This dehydration technique strongly accelerated the drying rate and maintained the antioxidant capacity and overall acceptance of roselle calyces powder. Reduced drying duration was noticed when high air temperature was applied. Filkova and Mujumdar⁶⁵ confirmed that temperature was not significantly effected at lower feed rate versus low drying rate. Maskat et

al.²⁹ examined the influence of input temperature (150, 160 and 170°C) and input productivity (280, 350 and 420 ml/h) on the most roselle powder characteristics during spray dehydration. There was significant impact of input temperature and input productivity on recovery, moisture content and overall acceptance. The powder yield was higher with high feed rate but lower with accelerated inlet temperature. However, feed rate did not cause any detrimental effect on the anthocyanin content in roselle powder. Esteban et al.⁶⁶ evaluated the effect of maltodextrin (10-20%), input temperature (130-150 °C), output temperature (75- 85°C) and spraying speed (22,000–26,000 rpm). The optimal drying operating variable were noticed at 20% maltodextrin, 130°C input temperature, 75°C output temperature and 22,000 rpm spraying speed. Audirene et al.⁶⁷ optimized the spray dehydration of jussara pulp with the support of modified starch and whey protein concentrate by input temperature from 140°C to 200°C. The input temperature 170°C was recommended. Bednarska and Janiszewska-Turak (68) demonstrated the role of input temperature (160°C, 200°C); maltodextrin and arabic gum as wall material on anthocyanins, polyphenols and physical powder characteristics received after spray dehydriaon. The mixtures of arabic gum: maltodextrin showed a huge potential to obtain an excellent chokeberry powder with high amount of anthocyanin and polyphenol. In another report, high quality of powder was received when outlet air temperature was in the range of 100-110°C⁶⁹. High temperature and short time were commonly applied to preserve unique property of anthocyanin⁷⁰. Temperature was not significantly effective at low feed rate⁷¹.

Table 4. Influence of feed rate (ml/min) on physicochemical property of *Hibiscus sabdariffa* powder

Feed rate (ml/min)	8	10	12	14	16
Bulk density (g/ml)	0.93±0.00 ^a	0.84±0.03 ^{ab}	0.76±0.01 ^b	0.65±0.02 ^{bc}	0.53±0.00 ^c
Solubility (%)	90.04±0.03 ^a	83.54±0.00 ^{ab}	78.25±0.02 ^b	70.32±0.00 ^{bc}	63.40±0.03 ^c
Total phenolic (mg GAE/ 100 g)	484.12±1.37 ^{bc}	496.33±1.24 ^b	519.14±1.08 ^a	503.23±1.16 ^{ab}	461.08±1.02 ^c
Total flavonoid (mg QE/ 100 g)	201.57±0.12 ^{bc}	219.57±0.18 ^b	268.65±0.10 ^a	247.60±0.17 ^{ab}	169.87±0.11 ^c
Anthocyanin (mg/100g)	164.29±0.13 ^{bc}	187.42±0.07 ^b	230.07±0.13 ^a	211.50±0.09 ^{ab}	134.22±0.15 ^c

Figures are the mean of three replications; Figures in row followed by the same letter/s are not differed significantly (α = P=0.05).

CONCLUSION

Roselle (*Hibiscus sabdariffa* L.) calyx was brilliant red in colour due to the present of anthocyanins. The consumption of roselle calyx powder could promote health benefits. This research successfully investigated some technical parameters for spray drying roselle calyx extract into dried powder. Sugar alcohols (mannitol, sorbitol, isomalt, xylitol, erythritol), carrier agents (maltodextrin, gum arabic, glutinous starch, whey protein concentrate, carboxymethyl cellulose), operating variables (inlet/ outlet air temperature, feed rate) significantly affected to the physicochemical attributes of the roselle dried powder. The best drying parameters found in the present study were noticed at 8% isomalt as supplement; 12% whey protein concentrate as carrier agent; inlet/ outlet air temperature 140/85°C/°C and feed rate 12 ml/min as the main operating parameters.

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DATA AVAILABILITY

All datasets generated or analyzed during this study were included in the manuscript.

ETHICS STATEMENT

This article does not contain any studies with human participants or animals performed by the author.

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