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Research Article

Preparation and Optimization of a Novel Tartary Buckwheat Instant Tea Powder by Spray Drying

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Abstract

In this work, the spray-drying conditions for the preparation of tartary buckwheat (TB) insant tea powder (TB-ITP) were optimized using response surface methodology (RSM). Firstly, the alcohol-soluble and water-soluble active components of TB were extracted using ultrasonic assisted extraction technology and then were spray-dried using β-cyclodextrin (β-CD) as a carrier to prepare TB-ITP. The parameters for spray drying, such as inlet air temperature, the feed flow rate and dry air flow rate were optimized using a Box–Behnken design, the yield was analyzed as a response. The morphology, feel, solubility bulk density, physical stability of the spray-dried TB-ITP were characterized. Inlet air temperature 147.2 ^{o}C, the feed flow rate was 3.75 mL / min and the dry air flow rate was 65.6 L / min as the optimum conditions. Under optimum conditions the yield reached a maximum of 45.3%, which provided an efficient method for the production of TB-ITP. After 3 months of storage, the appearance, moisture content, bulk density and solubility of TB-ITP were slightly changed, indicating a good stability under sealed condition.

Keywords: tartary buckwheat; tea; spray drying

Introduction

Tea is the second most commonly consumed drink in the world, which is of great popularity due to its beneficial medicinal properties, economical and safe characteristics[1-3]. As one type of tea products, instant tea can satisfy many aspects of demands of the human life and therefore become one of the important components of human life in the modern time. Instant tea, also known as tea essence, is manufactured from processed leaves. It is a kind of convenient solid beverage like powdered or fragmented or granular instant tea which is prepared by filtering, concentrating and drying a concentrated water extract of tea[4]. Nowadays, high cost of production and impairment of the organoleptic properties of tea are main problems associated with instant tea manufacture[5], quality control of tea becomes more and more important, in general, active ingredients are analyzed as the important quality factors for tea leaves. These constituents are mainly responsible for the characteristic astringent and bitter taste of tea brews[6]. Heavy metal in instant tea is considered negative because of possible health problems associated with excessive intake of them[7]. Homology of medicine and food has also gradually attracted into people's attention, the development of functional health instant tea beverages become a new trend.

The development of instant tea is rapid, nowadays, instant tea is a popular, widely accepted, pleasant, economical and safe drink that is enjoyed every day by millions of people across all continents[8]. Its popularity is due mostly to its availability of varieties of tastes. Moreover, instant tea is also consumed for health reasons because it contains large quantity of tea polyphenols, saccharide, protein, caffeine, vitamins, inorganic elements and trace elements such as physiological active element[9]. Besides,it has other advantages, such as, easy to drink (add water to drink); no garbage; relative volume (or weight) is small; easy to transport; does not contain harmful substances[10]; rich in nutrients and flavor substances; It has all the useful effects of tea on the human body.

Tartary buckwheat (*Fagopyrum tataricum* Gaertn.) is increasingly considered as an important functional food material because of its rich nutraceutical compounds[11]. Reserve starch is the major component of tartary buckwheat seed. Its main active ingredients are flavonoids[12] and it is rich in oleic acid, vitamins, niacin, and dietary fiber [13-16]. At the same time, tartary buckwheat (*Fagopyrum tataricum* Gaertn) is a minor crop belonging to the Polygonaceae family [17]. In addition, tartary buckwheat (*Fagopyrum tataricum*) contains a range of nutrients including bioactive carbohydrates and proteins, polyphenols, phytosterols, vitamins, carotenoids, and minerals[18]. The unique composition of tartary buckwheat contributes to their various health benefits such as anti-oxidative, anti-cancer, anti-hypertension, anti-diabetic, cholesterol-lowering, and cognition-improving[19-22] .

In this paper, the alcohol-soluble and water-soluble active components of TB were extracted using ultrasonic assisted extraction technology and embed them with β-CD, then spray dried to prepare tartary buckwheat instant tea powder (TB-ITP).

The spray drying procedures in preparation of TB-ITP were developed and the process parameters were optimized. In the preliminary studies, spray drying such as inlet air temperature, feed flow rate and drying air flow speed would strongly influence moisture content and yield of tea powders. Therefore, to optimize the experiment and analyze the interactions between factors, the model by Box-Behnken design was established and assessed. Besides, in order to ensure the quality of instant tea, the sensory indexes, active ingredient index, physicochemical index, metal indicators and health indicators were evaluated in this paper.

Materials and methods

Materials

Tartary buckwheat was purchased from ChaQiankun Co., Ltd. (Zhejiang, China). Reference standards of flavonoids (99.6%) were provided by the National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China).

Sodium nitrite, sodium hydroxide and ethanol were provided by Beichen Founder Regent Factory (Tianjin, China). Aluminum nitrate was provided by Fuchen Chemical Regents Factory(Tianjin, China). Cyclodextrin was provided by Bodi Chemical Co., Ltd. Distilled water was used throughout the study.

Main Instrumentation

Determination of flavonoids was performed on a UV spectrophotometer (Puxitongyong Instrument Co., Ltd, Beijing, China). The stirring experiments were performed using a digital constant temperature magnetic stirrer (HJ-6A, Baita Xinbao Instrument Factory, Jiangsu,China). The spray drying experiments were performed on a YC-015 experimental spray drier (Yacheng Experimental Co. Ltd., Shanghai, China). Electron microscope photograph were taken by a scanning electron microscopy (JSM-7500F, Japan Electronics Co., Ltd., Japan). The process of evaporative concentration uses a rotary evaporator (RE-52A, Yarong Biochemical Instrument Factory, Shanghai, China).

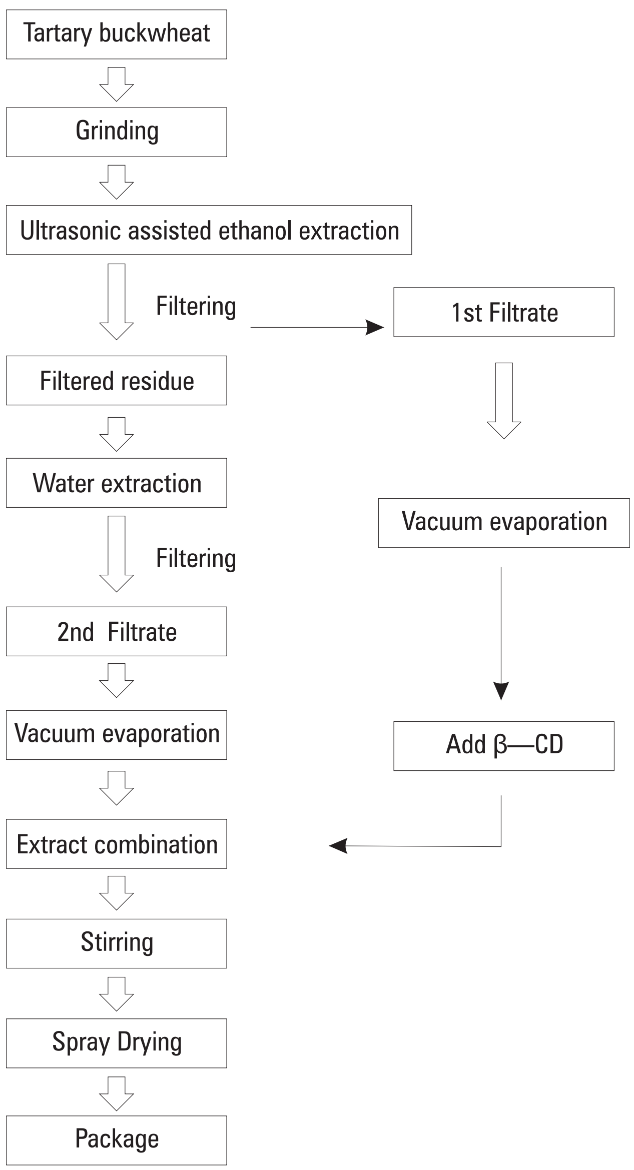


Figure 1: The production flowchart of TB-ITP

Preparation of TB tea condensated extracts

The TB was crushed and screened through 20 mesh sieve. 20 g crushed tartary buckwheat powder were accurately weighed and put in a 500 ml beaker, 300 ml ethanol were added under ultrasonic assisted extraction for about 30 min, then the solution was filtrated and the residue was added 300 ml distilled water at 80 °C for 2 h and then filtered. The residue is dried and weighed to calculate the yield. The ethanol extract and water extract were concentrated by rotary evaporation at 60 °C and 80 °C, respectively. And then the extracts were mixed. In order to retain the aroma components of tea, enhance the humidity resistance and storage security of instant tea powder, the appropriate amount of β-CD were added in water-ethanol condensated extracts at a certain temperature (50 °C - 60 °C) under a magnetic stirring for 30 min. Then the solution was spray-dried using a YC-015 experimental spray drier with the appropriate process parameters. The TB-ITP powders were collected for further study. Figure 1 showed the production process of TB-ITP.

Experimental design

A three-factor, three-level Box-Behnken design is suitable for exploring the effects of the processing variables on the quality of spray dried TB-ITP with Design Expert® 8.0.7.1 (Stat-Ease Inc., Minneapolis, Minnesota, USA).

The independent and dependent variables are listed in Table 1 along with their low, medium and high levels. The independent variables are inlet air temperature (A), feed flow rate (B), air flow speed (C) and the product yield (Y) is selected as the dependent variable. The values of product yield for each trial of the experimental design are shown in Table 2.

Table 1: Experimental parameters for Box–Behnken design

Factors	Process parameters	Levels		
		Low (-1)	Medium (0)	High (+1)
	Independent variables			
A	inlet temperature (°C)	135	145	155
B	feed flow rate (ml/min)	1	3	5
C	air flow speed (L/min)	50	65	80
	Dependent variables			
Y	Yield (%)			

Evaluation of spray-dried TB-ITP

Sensory evaluation

Sensory characteristics of TB-ITP samples were evaluated by 10 healthy volunteers. each sample (0.5g) dissolved with 100ml of 80 °C hot water in a transparent glass bottle.

The appearance and smell of each sample was observed first and then the smell was evaluated, each volunteer taste 5ml sample solution and kept in the mouth for 10 sec. The score was recorded and each evaluation was repeated twice.

Determination of product yield

The yield of TB-ITP was calculated as ratio of the weight of the resultant powder after spray drying in collecting bottle and the weight of all solids in the feeding suspension, any powder adhering to the walls of drying chamber or cyclone were not considered.

It is calculated as follows:

$\text{Product yield} = m / m_0 \times 100\%$

m: the weight of spray-dried powder

m_0:the weight of used raw material before spray drying

Determination of solubility and pH

5 g TB-ITP sample was accurately weighed and put into a 50ml beaker, then 30 ml of deionized water was added. After that the beaker was under ultrasonic for 5 min at room temperature. The

solution was completely transferred to a 50 ml volumetric flask and diluted with deionized water to 50 ml, then 15 ml solution was centrifuged for 15 min at 4000 r/min, the supernatant, was fully transferred into the dish, then was put into 105 °C oven drying to constant mass. Solubility was characterized by Drying Matter Solubility Index (DSI) . DSI % = The quality of solids in the supernatant fluid / the quality of sample × (1 – the water content of TB-ITP) ×100%

For pH determination, 1g TB-ITP was dissolved in a beaker with 200ml 37 °C water and pH of the solution was determined.

Determination of moisture

Moisture was determined in accordance with the Chinese Pharmacopoeia (2015 edition).The moisture content was determined by drying of 5g samples at 100～105 °C in an oven until a constant weight was obtained.

The moisture content was calculated as:moisture content $=A_1/A_2\times 100\%$

A_1: the loss weight of water, A_2: weight of powder sample, all experiments were performed in triplicate.

Determination of hygroscopicity

The instant tea powder was hot pressing packaged with aluminum foil bag, After that place it into the dryer under 75% relative humidity at 60 °C for ten days. Percentage of moisture absorption was calculated.

Morphology of TB-ITP

The shape and surface morphology of TB-ITP were evaluated by a scanning electron microscopy (SEM). The TB-ITP was initially fixed on stubs using a double-sided sticky and coated with gold under a high-vacuum atmosphere and subsequently observed by SEM (JSM-7500F, Japan Electronics Co., Ltd., Japan) at an acceleration voltage of 10 kV.

Physical stability

Physical stability is a crucial issue in formulating dry powders. TB-ITP was sealed in vials and stored for 3 months at 4 °C in a refrigerator. The stability study was examined after 3 months. Samples were taken to determine moisture, bulk density and solubility again.

Results and discussion

Design of experiments

A total of 17 experiments were carried out to study the effect of experimental parameters on the product yield and moisture content (Table 2). A second-order polynomial equation was used to express the response variable as a function of the independent variable as follows:

$$Yield = 45.26 + 2.83\times A + 2.08\times B - 1.0\times C - 1.65\times A\times B + 4.65\times A\times C + 1.0\times B\times C - 5.38\times A^2 - 2.33\times B^2 - 4.98\times C^2$$

Where Y represents the product yield, A, B, C are inlet air temperature, feed flow rate, air flow speed, respectively.

Table 2: Experimental runs, independent variables, and measured response of the Box-Behnken Design

	A	B	C	Y
Run	**Inlet temperature (°C)**	**feed flow rate (ml/min)**	**Air flow speed (L/min)**	**Yield (%)**
F1	135	5	65	41.8
F2	145	3	65	46.4
F3	145	3	65	45.2
F4	145	3	65	45.9
F5	145	3	65	45.1
F6	145	1	50	40.1
F7	145	1	80	35.4
F8	145	3	65	43.7
F9	145	5	50	38.5
F10	145	5	80	37.8
F11	155	1	65	36.6
F12	155	3	80	43.2
F13	155	5	65	41.2
F14	155	3	50	35.2
F15	135	1	65	30.6
F16	135	3	50	35.9
F17	135	3	80	25.3

Response surface analysis

Product yield

The effect of different independent variables on the product yield was shown in Figures 1a-1c, it could be seen that increasing feed flow rate could increase the yield of spray drying. Figure 1a and 1b showed that the increase of dry temperature improved the product yield first and then decreased the yield. In our study, the outlet air temperature is mainly dependent on the feed rate and less on the aspirator rate, air humidity and spray air flow.

The outlet temperature was an important factor on the product quality. At first, with the outlet temperatures increased the moisture content of the powder reduced, the yield of spray drying increased as the outlet air temperature increased. But higher outlet temperatures resulted in a lower yield, it can be attributed to partially sticking of the insufficiently dry particles on the dryer walls at higher temperatures.

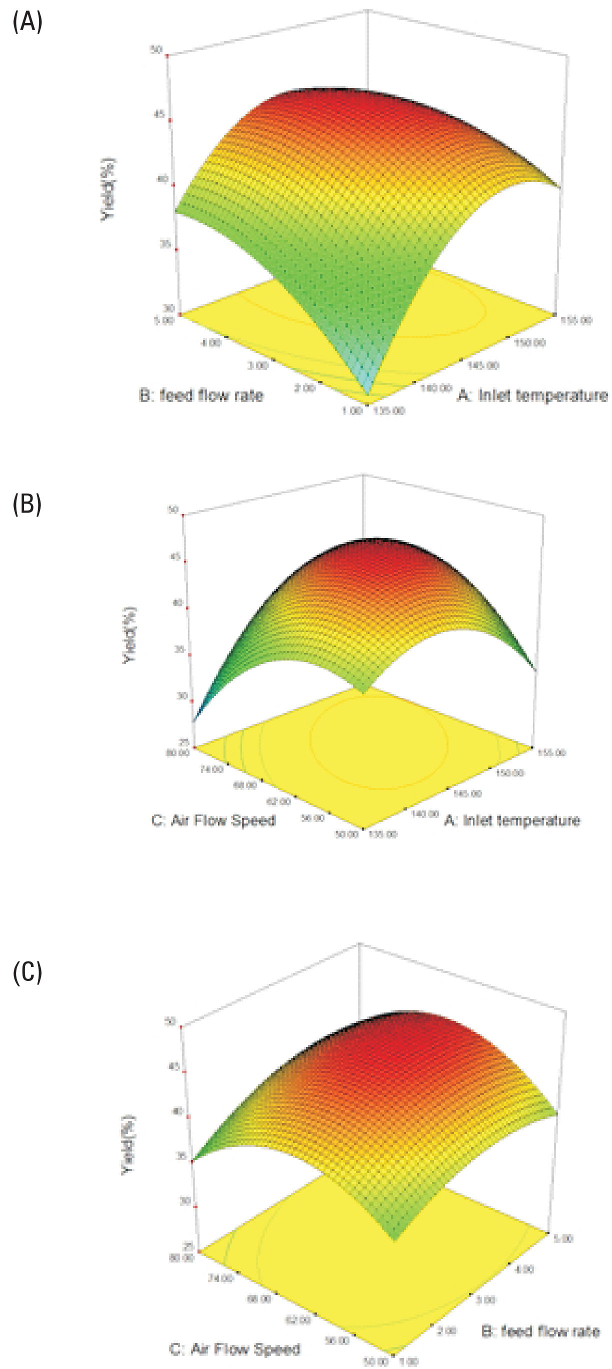


Figure 2: Response surface plots for product yield. (A) inlet temperature, (B) feed flow rate, (C) air flow speed

Optimization of spray-dried TB-ITP

The responses of the optimized formulations were evaluated to ensure the product yield and moisture content. Table 3 presented the composition, predicted and observed responses of the optimized formulation. Results showed that the observed values of the prepared optimized spray-dried formulation were mostly similar with predicted values.

Table 3: Composition, predicted, and observed responses of the optimized spray-dried formulation

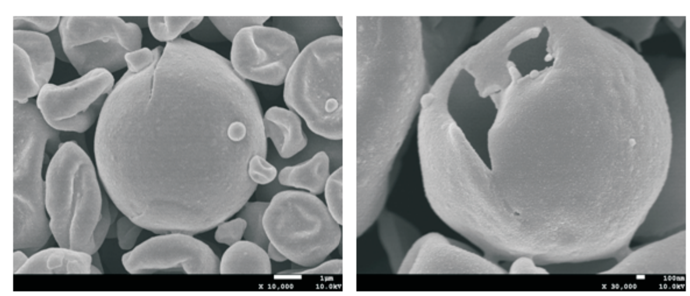
Variables	Values	Responses	Predicted values	Observed values
Inlet temperature (°C)	147.2	Yield (%)	45.9%	45.3%
Feed flow rate (mL/min)	3.75			
Air flow speed (L/min)	65.6			

Physicochemical properties of TB-ITP

The physicochemical properties of the spray dried TB-ITP were investigated. Water solubility of TB-ITP was found to about 97.8%. After 3 months of storage, the appearance, moisture content, bulk density and solubility of TB-ITP slightly changed with increased storage, indicating a good stability in sealed aluminum coil bag.

From Figure 3A, it was found that the powders were irregularly spherical shaped particles, not aggregated together and some roughness and shrinkages on the surface. The SEM micrograph also showed some holes or cracks in the surface of powders (Fig 3B). When the droplets were aerosolized by spray drying, the moisture was rapidly evaporated from the spherical droplet when it came into contact with hot air, and formed hollow spherical droplets. In Figure 4, the spherical droplet rupture shows that it is hollow inside.

The sensory results of TB-ITP are shown in Table 4, it was found that TB-ITP, the TB-ITP exhibited satisfying appearance, smell and taste attributes, the standing time did not significantly influenced the sensory results. After standing for over 30 min TB-ITP still kept a perfect appearance and taste.



A. B.

Figure 3: SEM images of the optimized spray-dried TB-ITP

Table 4: Sensory evaluation of TB-ITP (n=4)

Time(min)	Score		
	Appearance (30)	Smell (30)	Taste (40)
5	27.0±2.4	28.3±2.4	26.8±0.5
15	26.0±1.2	27.8±2.6	26.8±1.3
30	27.5±2.1	28.0±2.4	25.0±2.2
60	26.0±0.8	27.0±2.4	25.3±0.5

Conclusion

In this study TB-ITP was successfully spray dried from the TB extraction solution with the adding of β-cyclodextrin. The spray drying conditions were optimized using process yield as a response. Furthermore, other characteristics such as the morphology, taste, solubility bulk density and physical stability of TB-ITP were also conducted. Experimental results proved that the optimum process conditions for obtaining spray-dried TB-ITP were inlet air temperature of 147.2 ºC, a feed flow rate of 3.75 mL/min and a drying air flow speed of 65.6 L/min. The TB-ITP developed using the optimized spray-drying process showed a small particle size and uniform particle shape. TB-ITP had the good solubility in water with good stability. Therefore, spray-dried TB-ITP had excellent physical properties and was potentially used as instant tea for commercial application in food and nutraceutical industries.

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Conflict of Interest: The authors confirm that this article content has no conflict of interest.

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