Optimization of Process Conditions for *Batuan* [*Garcinia binucao* (Blanco) Choisy] Fruit Powder Production

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Abstract

The study determined the optimum process conditions to produce *batuan* fruit powder using combined Response Surface Methodology (RSM) and desirability function. The factors considered were sodium metabisulfite (SMS) concentration and drying temperature. Two-factor ANOVA revealed the significant responses among the physicochemical (bulk density, titratable acidity, total soluble solids, whiteness index) and functional (antioxidant activity, total phenolics, water absorption index, water solubility index) characteristics. The response that was not significant in the model was also identified (pH). Based on the results, the optimum drying temperature and SMS concentration were found to be 50.0 °C and 106 ppm, respectively. The powder was produced using the predicted optimum conditions and was analyzed for its physicochemical, functional, and sensory properties.

[Keywords] *batuan* fruit, *batuan* fruit powder, response surface methodology, sodium metabisulfite, drying

I Introduction

Tamarind is considered as the most popular souring agent in Philippines and is used as a base in soup dishes such as sinigang. However, the supply of locally-available tamarind may not be able to meet the huge demand of consumers due to the increase in population. Philippines is continuously importing tamarind to meet the domestic needs (Valencia, 2013a; Reyes, 2000; Mojica, 2008), thus, the need for an alternative souring agent.

The potential of converting indigenous crops, such as *batuan* [*Garcinia binucao* (Blanco) Choisy] fruit that is popular in southern Philippines, into high-value products are currently observed by researchers (Ebert, 2014; Valencia, 2013b; Florido and Cortiguerra, 2003). Quevedo et al. (2013) reported that *batuan* fruit is nutritious and is also safe for consumption. Indeed, *batuan* fruit is a promising alternative souring agent.

Even if *batuan* is abundant, it has limited availability only from April to June. Thus, preservation of this fruit is necessary to make it available throughout the year, one of which is to convert it into powder.

There are many researches nowadays about production of powders including mango powder (Jaya and Das, 2005), date powder granules (Sablani et al., 2008), gac fruit aril powder production using spray drying (Kha et al., 2010), tamarind powder production by drum drying using maltodextrin and

arabic gum as adjuncts (Jittanit et al., 2011), comparison of qualities of tamarind powder using tray and drum dryers (Khuenpet et al., 2012), fiber-rich powder from dragon fruit or pitaya peel (Sengkhamparn et al., 2013), mango kernel flour production using cabinet dryer (Bawar et al., 2013), and spray-drying of soursop powder (Chang et al., 2018; Chang et al., 2019) among others. There are also published studies about *batuan* fruits' physicochemical properties, nutritional and sensory qualities (Quevedo et al., 2013), organic acid profile (Quevedo et al., 2017), and hydroxycitric acid content that is affected during processing (Bainto et al., 2018). Recent studies about *batuan* fruit processing were published by Belmes (2019), Dormido et al. (2019), and Lascano et al. (2019). However, there is limited published article about *batuan* fruit powder.

The production of the powder included drying, grinding, and other interventions such as inactivation of enzymes and addition of preservatives to ensure a high-quality product. Drying utilizing hot air was done to remove most of the moisture and to produce a powder with good flowability. Before drying, sodium metabisulfite (SMS), an anti-browning agent, was added to the fresh pulp. Therefore, this study was designed to establish the procedure to produce powder, a high-value product, from the pulp of the *batuan* fruit. Also, the optimum SMS concentration and drying temperature were determined based on the physicochemical and functional

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characteristics for *batuan* fruit powder processing.

II Materials and Methods

1. Preparation of *batuan* fruit powder

(1) Source of *batuan* fruits

Batuan fruits were bought from a local market in Bacolod City, Philippines and then shipped to the Institute of Food Science and Technology, University of the Philippines Los Baños for study. The fruits used in the experiment were immature (with characteristic green color and hard covering) and had medium-to-large size (about 3.7–5.5 cm in diameter) only (Fig. 1). The sizes that were used in the study were the 3 largest diameters which are medium to large.

(2) Preparation of frozen *batuan* fruits

Immediately when the fresh fruits were received, they were initially washed with tap water to remove adhering contaminants, then the whole fruits were disinfected by soaking in 10 ppm hypochlorous acid (HOCl) solution for 20 seconds to lessen the initial microbial load. The fruits were washed again in running potable water to remove excess chlorine on the fruits. Next, the fruits were packed in PE bags (about 10 kg per bag), then stored in a chest freezer at -20°C . Before processing into powder, the frozen fruits were thawed in running water for about 5 min and drained well.

(3) Pre-drying treatments

The whole fruits were cooked using a steamer for 20 min or longer until the color of the peel has completely changed from green to yellowish brown. Steaming was necessary to inactivate the enzymes and to soften the pulp for easier removal by the pulping machine. After steaming, the cooked fruits were immediately cooled with running water to stop overcooking. Then, the steamed fruits were fed through a pulping machine to separate the seeds and recover the pulp.

The recovered pulp was separated into 3 lots where each lot was treated with a stock solution of 10% (w/w) sodium metabisulfite (SMS). SMS was added to the pulp so that the pulp would contain 0 (control), 125 and 250 ppm concentration.

(4) Convective drying of *batuan* fruit pulp

The pretreated pulp was dried using a cabinet dryer that was fabricated and installed in the Institute of Food Science and Technology, UPLB. The pulp was laid on stainless steel trays layered with polyethylene to prevent the pulp from sticking onto the trays after drying. The thickness of the pulp on the trays was set at 3 mm maximum to allow faster drying. Three different drying temperatures (50, 60, 70 $^{\circ}\text{C}$) were employed in the study. The drying of the pulp was continued until the sample reached a moisture content of about 10.75 % (wet basis) such that the product became brittle in texture. The dried pulp was like thin, brittle, brown flakes were scraped using a spatula.

(5) Grinding and sieving

A grinder (Koi Platinum Edition, Koi, Philippines) was used to grind the dried pulp. The produced powder was sieved using 60-mesh USA sieve to obtain a finer powder (Fig. 2 (b)). Then the produced powder samples were immediately packed in glass bottles at room temperature for storage.

2. Physicochemical analysis

The *batuan* fruit powder samples were subjected to physicochemical analysis in terms of bulk density (BD), fineness modulus (FM), pH, titratable acidity (TA), total soluble solids (TSS), and whiteness index (WI).

(1) Bulk density (BD)

The powder sample was filled into a pre-weighed 50-mL graduated cylinder up to the 50-mL mark. No tapping or compression of the powder was done to avoid variation in the results. The values of BD were expressed in terms of g / mL:

$$BD = \frac{m_{sc} - m_c}{V_s} \quad (1)$$

where *BD* is the bulk density (g / mL), *m_{sc}* is the mass of sample and cylinder (g), *m_c* is the mass of cylinder (g), *V_s* is the volume of sample (mL).

Fig. 1 Relative sizes of *batuan* fruits used in the study

Fig. 2 Sieving of *batuan* powder using 60-mesh USA sieve showing (a) reject oversize and (b) product undersize

(2) pH

The pH of the *batuan* fruit powder was determined by using a pH pen (Eutech Instruments pH 2700, Eutech Instruments Pte. Ltd., Singapore) in a 1 : 9 ratio by mass (dilution factor of 10) mixture of *batuan* powder and distilled water.

(3) Titratable acidity (TA)

The *batuan* fruit powder was dissolved in freshly boiled and cooled distilled water at a ratio of 1 : 9 by mass (dilution factor of 10). The resulting solution was added with 2–3 drops of 1 % phenolphthalein indicator and titrated using 0.1 M NaOH solution up to faint pink endpoint. Then the %TA was calculated using the formula:

$$\%TA = \frac{V_t \times N_t \times W_{t_{acid}} \times DF}{V_s \times 10} \quad (2)$$

where %TA is the percentage of titratable acidity (g citric acid / 100 g sample), V_t is the volume of titrant used (mL), N_t is the N titrant, $W_{t_{acid}}$ is the eq. wt. acid (= 64.04 for citric acid), DF is the dilution factor, V_s is the volume of sample (mL).

(4) Total soluble solids (TSS)

The *batuan* fruit powder was dissolved in distilled water at a ratio of 1 : 9 by mass (dilution factor of 10). Using a refractometer (Cole-Parmer Refractometer EW-81150–32, Cole-Parmer Instrument Company LLC, USA), the degree Brix (°Bx) of the solution was read.

(5) Whiteness index (WI)

The color of the sample was measured using a chromameter (X-Rite Capsure RM200-PT01, X-Rite Inc., USA) as L (lightness), a (redness), and b (yellowness). The values of L , a , and b obtained were used to calculate the WI using the equation according to Hsu et al. (2003) and Bawar et al. (2013):

$$WI = 100 - \sqrt{(100 - L)^2 + a^2 + b^2} \quad (3)$$

3. Functional analysis

The powder samples were also analyzed for functional properties in terms of antioxidant activity, total phenolics, water absorption index (WAI), and water solubility index (WSI).

(1) Antioxidant activity

The antioxidant activity of the *batuan* fruit powder was determined based on its ability to scavenge stable DPPH. The sample was placed 50 mg in a test tube and then added with 5 mL of 80 % (v / v) methanol solution and mixed in a vortex mixer for 10 min. The mixture was then filtered (No. 1 filter paper, Whatman, UK) in a test tube and kept refrigerated until use. A 1-mL aliquot was obtained and added with 4 mL distilled water. Freshly prepared 1 mL of 1 mM methanolic DPPH solution was added. The solutions were allowed to stand for 30 min. The absorbance of the solutions (sample and blank) was read at 517 nm (Bawar et al., 2013; Murthy et al.,

2002 as cited in Veigas et al., 2007). The percentage scavenging activity of DPPH was computed using the equation:

$$\%DPPH_{sa} = \left(1 - \frac{A_{test}}{A_{blank}}\right) \times 100 \quad (4)$$

where %DPPH_{sa} is the percent scavenging activity of DPPH (%), A_{test} is the absorbance test sample, A_{blank} is the absorbance blank sample.

(2) Total phenolics

Folin-Ciocalteu Method was used to analyze total phenolics. The powder samples were diluted to 1000 µg / mL with 80 % methanol solution and then filtered using Whatman No. 1 filter paper to remove suspended solids (that may interfere during reading of absorbance using a spectrophotometer). Exactly 0.25 mL, each, of diluted samples and standard solutions (0, 40, 80, 100, 150 µg / mL gallic acid) was obtained and diluted with 3.5 mL distilled water. Then 0.5 mL of 50 % Folin-Ciocalteu reagent was added followed by 1 mL of 20 % Na₂CO₃ after 3 min. Then the samples were mixed and incubated in boiling water for 1 min to develop the blue color. Then absorbance of the samples was read at 685 nm. The total phenolics content (µg gallic acid equivalent / mg powder) of the samples was calculated based on standard curve of the standard solutions.

(3) Water absorption index (WAI) and water solubility index (WSI)

WAI and WSI were determined in triplicates following the method by Anderson (1982 as cited in Narbutaite et al., 2008). About 1 g of each sample was suspended in 6 mL of distilled water and stirred for 30 min at 30 °C. Then, the mixture was centrifuged at 4000 × g for 20 min. The supernatant liquid was poured into a dry 15-mL test tube and stored overnight at 110 °C to evaporate the water. The WAI and WSI were computed using following equations:

$$\%WSI = \frac{m_{ds}}{m_s} \times 100 \quad (5)$$

$$WAI = \frac{m_{sed}}{m_s} \quad (6)$$

where %WSI is the percentage of water solubility index (%), m_{ds} is the mass of dissolved solids in supernatant (g), m_s is the mass of sample (g), WAI is the water absorption index, m_{sed} is the mass of sediment (g).

4. Design of experiment and statistical analysis

All analyses were done in triplicates. The determined values were expressed as mean ± standard deviation. Data were analyzed using Analysis of Variance (ANOVA) to determine if the sample means significantly differed from one another, followed by Tukey's Honest Significant Difference (HSD) test to know which among the means were significantly different. ANOVA and HSD test were aided by Statistical

Analysis System (SAS Version 9, SAS Institute Inc., USA) computer software.

The experimental design was based on complete factorial design (CFD) such that there were 2 factors (SMS concentration, drying temperature) and 3 levels for each factor, hence, $3^2 = 9$ treatments. There were 9 responses considered from physicochemical and functional properties (bulk density, pH, titratable acidity, total soluble solids, whiteness index, antioxidant activity, total phenolics, water absorption index, water solubility index). Combined methods of response surface methodology (RSM) and desirability function were applied to the experimental data to generate regression models and calculate the value of desirability following the procedure of Design-Expert (Version 9.0.3.1, Stat-Ease Inc., USA) computer software. From the models, a contour plot was created for each response to assess the effect of the 2 factors. The regression models (of the responses given the 2 factors) were screened using Two-factor ANOVA by calculating (1) regression coefficient, R^2 , to evaluate the fitness of each model and (2) p -value to determine whether the models were significant or not. Then, the optimum SMS concentration and drying temperature was calculated using the software. The usefulness of the model to predict the optimum condition was measured in terms of an overall desirability function, D . Verification was also performed to determine if the actual and experimental values agree with one another.

5. Analysis of optimized *batuan* fruit powder

The *batuan* fruit powder was produced following the predicted optimum conditions and analyzed of its physicochemical, functional, and sensory properties.

(1) Physicochemical and functional analysis

The optimized *batuan* fruit powder was analyzed again in terms of BD, pH, TA, TSS, WI, antioxidant activity, total phenolics, WAI, and WSI following the above-mentioned protocols.

(2) Proximate analysis

In addition, proximate analysis (moisture content, ash, crude protein, crude fat, crude fiber, total carbohydrates) was done to further characterize the optimized *batuan* fruit powder. The procedure based on AOAC (2000) was followed.

(3) Sensory evaluation

Sensory analysis of the optimized sample was also done using Quality Scoring which was performed by 10 semi-trained panelists. The parameters evaluated were color, aroma, off-odor, and general acceptability. The test used a 15-cm line for each parameter with increasing intensity from left (score of 0) to right (score of 15). The color was assessed as “light brown” (0) to “dark brown” (15) with sample pictures showing standard colors of light brown and dark brown to the judges

Fig. 3 *Batuan* fruit powder produced at varying temperature and SMS concentration

for reference. To measure aroma, from “weak” (0) to “strong” (15), a sample of fresh and puréed *batuan* fruit was used as reference. Off-odor was gauged from “not perceivable” (0) to “perceivable” (15). General acceptability was determined from “not acceptable” (0) to “acceptable” (15).

III Results and Discussion

The effects of varying SMS concentration (0, 125, 250 ppm) and drying temperature (50, 60, 70 °C) on the quality of *batuan* fruit powder (Fig. 3) were evaluated.

The intensity of the brown color increased with temperature and decreased with SMS concentration; similar result was observed by Ajaykumar et al. (2012) in dried green chilli powder. However, this trend was observable only at 70 °C. At 50 °C, the lightest brown color was noticed at 125 ppm SMS. At 60 °C, there was no significant difference between the observed colors of the samples. Hence, chromameter was used to accurately measure the color of the samples in terms of WI.

1. Effect of SMS concentration and drying temperature on the physicochemical properties of *batuan* fruit powder

The results of the effects of SMS concentration and drying temperature on the physicochemical properties of the powder were summarized in Table 1 and Fig. 4.

(1) Bulk density (BD)

The bulk density accounts for the true volume occupied by the product and the volume of the voids or spaces between the particles. Higher bulk density is favorable because at a certain mass of product, less volume is occupied and therefore the

Table 1 Physicochemical properties of *batuan* fruit powder at varying SMS concentration and drying temperature

Physicochemical Property	SMS Concentration (ppm)	Temperature (°C)		
		50	60	70
Bulk density, BD (g / mL)	0	0.666 ± 0.025 ^a	0.620 ± 0.005 ^a	0.648 ± 0.007 ^a
	125	0.654 ± 0.034 ^a	0.500 ± 0.002 ^b	0.637 ± 0.008 ^a
	250	0.625 ± 0.030 ^a	0.416 ± 0.003 ^c	0.613 ± 0.004 ^a
pH	0	3.40 ± 0.00 ^a	3.33 ± 0.06 ^a	3.40 ± 0.00 ^a
	125	3.40 ± 0.00 ^a	3.37 ± 0.06 ^a	3.40 ± 0.00 ^a
	250	3.40 ± 0.00 ^a	3.37 ± 0.06 ^a	3.37 ± 0.06 ^a
Titratable acidity, TA (g citric acid / 100 g)	0	24.38 ± 1.29 ^a	25.62 ± 1.43 ^a	20.87 ± 0.36 ^b
	125	23.35 ± 0.72 ^a	25.41 ± 1.24 ^a	21.49 ± 0.95 ^b
	250	23.35 ± 1.79 ^a	24.17 ± 1.24 ^a	21.49 ± 0.95 ^b
Total soluble solids, TSS (°Bx)	0	4.60 ± 0.00 ^a	4.67 ± 0.12 ^a	5.00 ± 0.00 ^b
	125	4.60 ± 0.00 ^a	4.73 ± 0.12 ^a	5.00 ± 0.00 ^b
	250	4.87 ± 0.12 ^a	4.53 ± 0.12 ^a	5.07 ± 0.12 ^b
Whiteness index, WI	0	44.59 ± 2.03 ^a	44.61 ± 2.06 ^a	36.81 ± 4.55 ^b
	125	46.94 ± 0.00 ^a	44.32 ± 1.02 ^a	41.34 ± 0.10 ^b
	250	46.94 ± 0.00 ^a	43.85 ± 1.94 ^a	39.33 ± 3.58 ^b

Mean values of the same superscript for all treatments for each physicochemical property are not significantly different at $p \leq 0.05$, HSD.

transport of the product from one place to another is easier.

Lower values of BD were observed at 60 °C, and SMS concentrations of 125 and 250 ppm; other values were not significantly different from each other (as shown by the superscripts in Table 1). There was also an observed negative correlation between the BD and the SMS content especially at 60 °C. Increasing the drying temperature may cause further shrinkage of the pulp during drying as the volatile substances escaped from the pulp so that the resulting powder would be more compact. The decrease in bulk density at greater temperature was reported by Kha et al. (2010) in spray drying of *gac* fruit aril powder. On the other hand, higher amount of SMS may result in better protection of the pulp from oxidation where the pulp would maintain its integrity or structure, but the effect of SMS on the BD became pronounced only at 60 °C (Table 1). However, there is little or no research that could verify the role of SMS in changing the BD of a powder.

(2) pH

The pH is an important property for the *batuan* fruit powder as a souring agent. The pH may determine whether microorganisms would survive in the sample or not, or what group of microorganisms may grow. Moreover, the combined effect of low pH and low water activity of the dried product further controlled growth of any type of microorganism (Leistner, 2000).

Lower pH values were seen at 60 °C, however, the differences between pH values were not significant based on Two-factor

ANOVA at 5 % level of significance.

(3) Titratable acidity (TA)

The TA is a better measurement of the acidity than pH. Lower pH may measure the concentration of hydrogen ions (and consequently the amount of dissociated acids) but TA accounts all the acids present whether dissociated or not. The TA was expressed as percent citric acid because it is the predominant acid present in the *batuan* fruit (Quevedo et al. 2017).

The changes in TA may be primarily due to temperature only. Moreover, at 60 °C, highest values of TA were seen. However, statistical analysis at 5 % level of significance revealed that there were no significant differences between the TA values at 50 and 60 °C. Varying the SMS concentration did not have significant effect on the TA due to volatilization of SMS at high temperature during drying. Nonetheless, higher TA values may be observed between 50 and 70 °C. At 70 °C, low values of TA were observed due to deteriorative reactions such as oxidation that may have occurred for some of the acids (i.e., ascorbic acid, hydroxycitric acid) at higher drying temperature and prolonged period of drying (Bainto et al., 2018; Ding et al., 2017; Oliveira et al., 2015).

(4) Total soluble solids (TSS)

Based on the results, TSS generally increases with temperature although the change in TSS was significant only at 70 °C, possibly due to degradation of some components such as pectin and dietary fiber which form smaller units that are

Fig. 4 Contour plots of physicochemical properties of *batuan* fruit powder versus drying temperature and SMS concentration

more water-soluble which were observed by Garau et al. (2007) in orange fruit, and de Roeck et al. (2008) in carrot tissue. On the other hand, the SMS did not play a role in varying the TSS of the powder (same superscripts as SMS concentration increases at constant drying temperature in Table 1).

(5) Whiteness index (WI)

The values of WI range from 0 to 100 such that lighter samples have WI values approaching 100. The suggestion by Ajaykumar et al. (2012) to blanch and add sulfite to the samples was followed, and therefore, the decrease in browning (in terms of WI) of the samples was expected with increasing concentrations of SMS as anti-browning agent.

Generally, WI decreased when the drying temperature increased while the WI slightly increased when SMS concentration became greater. However, based on statistical analysis at $p \leq 0.05$, even though the model is significant (Table 3) and the lack of fit is not significant, only the drying temperature is a significant factor that affects the WI. Nevertheless, the combined effects of temperature and SMS concentration may result in an optimum WI at lower

temperature and higher SMS concentration in Fig. 4. The decrease in WI at increasing temperature may be due to increased rate of browning reactions at higher temperature. On the other hand, increasing the SMS content did not significantly affect the color even with the ability of SMS to inhibit browning reactions due to volatilization of SMS during drying.

2. Effect of SMS concentration and drying temperature on the functional properties of *batuan* fruit powder

The effects of SMS concentration and drying temperature on the functional properties of the powder were summarized in Table 2 and Fig. 5.

(1) Antioxidant activity

From the contour plots (Fig. 5), higher antioxidant activity values were achieved at lower temperature and higher SMS content. The heat-sensitive antioxidants present in the powder, such as phenolics and ascorbic acid, were easily oxidized at higher temperature. A study by Ahmed et al. (2010) revealed that for sweet potato flour, SMS was able to protect phenolics

Table 2 Functional properties of *batuan* fruit powder at varying SMS concentration and drying temperature

Functional Property	SMS Concentration (ppm)	Temperature (°C)		
		50	60	70
Antioxidant activity (% DPPH-scavenging activity)	0	26.04 ± 0.80 ^a	23.99 ± 3.21 ^b	21.66 ± 0.56 ^c
	125	25.05 ± 1.74 ^a	27.67 ± 1.72 ^b	19.53 ± 1.29 ^c
	250	30.29 ± 1.07 ^a	21.30 ± 2.33 ^b	19.04 ± 0.86 ^c
Total phenolics (µg GAE / mg)	0	11.93 ± 2.65 ^a	9.82 ± 2.50 ^a	8.24 ± 3.33 ^b
	125	14.36 ± 1.13 ^a	22.18 ± 3.68 ^a	5.34 ± 0.83 ^b
	250	12.35 ± 0.98 ^a	12.89 ± 0.95 ^a	7.61 ± 0.80 ^b
WAI (g sediment / g)	0	3.394 ± 0.188 ^a	3.617 ± 0.271 ^a	3.464 ± 0.227 ^a
	125	3.449 ± 0.060 ^a	4.081 ± 0.018 ^a	3.087 ± 0.056 ^a
	250	3.376 ± 0.156 ^a	4.357 ± 0.410 ^a	3.210 ± 0.038 ^a
WSI (g dissolved solids / 100 g)	0	24.02 ± 1.34 ^a	14.23 ± 0.41 ^c	23.29 ± 1.66 ^a
	125	18.98 ± 0.75 ^b	14.46 ± 0.37 ^c	26.20 ± 1.49 ^a
	250	15.92 ± 1.83 ^b	14.42 ± 2.37 ^c	23.85 ± 1.45 ^a

Mean values of the same superscript for all treatments for each functional property are not significantly different at $p \leq 0.05$, HSD. For antioxidant activity, values were compared with standards 1 mM BHA (10.90 ± 1.93^d) and 1 mM ascorbic acid (15.43 ± 3.57^e). GAE: gallic acid equivalent

Fig. 5 Contour plots of functional properties of *batuan* fruit powder versus drying temperature and SMS concentration

and vitamin C so that the samples treated with SMS had higher total phenolics and vitamin C than the control. Also, increasing the drying temperature from 50 to 60 °C resulted in decreasing concentrations of total phenolics and vitamin C (Morgan and Field, 1929). However, for *batuan* fruit powder, statistical analysis showed that only the temperature, not the SMS concentration, had a significant effect on the antioxidant activity of the *batuan* fruit powder.

(2) Total phenolics

The total phenolics content contributed to the antioxidant activity of a product. The values of total phenolics were observed to be higher at lower drying temperature since the phenolics were heat sensitive (Guiné et al., 2015). The effect of SMS on the total phenolics was not significant due to volatility of SMS at higher temperature so that the latter just escaped from the pulp during drying.

(3) Water absorption index (WAI)

WAI indicates the ability of the powder to absorb water due

to the hydrophilic groups present that hold water (Narbutaite et al., 2008).

From Table 2, the values of WAI of the samples were not significantly different. Nevertheless, the quadratic model to express WAI as a function of SMS concentration was significant so that the model was still useful for optimization. The increase in WAI from 50 to about 60 °C could be due to partial gelatinization of starch and protein resulting in increased water uptake. However, above 60 °C, there was higher rate of vaporization of liquids resulting in shrinkage of the polar sites and then poor absorption of moisture upon rehydration. Gunaratne and Hover (2002 as cited in Ahmed et al., 2010) explained that the difference in WAI could be due to variation in the degree of engagement of hydroxyl groups to form hydrogen bonds between starch chains, and loss of starch crystalline structure.

(4) Water solubility index (WSI).

WSI determines the mass of polysaccharides released from the powder granules upon addition of excess water (Yousf et al., 2017). However, since Quevedo et al. (2013) reported that *batuan* contains low amount of starch and protein (values not reported), there might be water-soluble substances other than starch that were released from the granules during the analysis.

Lower values of WSI were seen at 60 °C due to the semi-crystalline structure of starch and formation of hydrogen bonds between starch molecules. Above 60 °C, the heat caused the starch molecules to swell and expose the hydrophilic groups, thereby, increasing the solubility of the powder in terms of WSI (Eliasson and Gudmundsson, 1996 as cited in Ahmed et al., 2010). At 50 °C, greater WSI values were obtained because this was where 48 h was needed to dry the pulp. Moreover, the long drying time favored the swelling and exposure of hydrophilic groups of the starch molecules

even at a slower rate.

3. Optimum conditions for *batuan* fruit powder

Based on the physicochemical and functional analyses, the optimum drying temperature and concentration of SMS was determined using Design-Expert. During optimization, the ranges of factors drying temperature and SMS concentration were set at 50–70 °C and 0–250 ppm, respectively. The results of ANOVA of the responses were listed in Table 3.

The goal “maximize” or “minimize” was selected. For example, “maximize” was chosen for whiteness index since maximum value implied lightest brown color. None of the responses had a goal “minimize”. On the other hand, there was no goal for pH because the fit model was found to be not significant in the first place.

The importance of a criterion was described in terms of a rating from 1 to 5 (where “5” is of highest importance). The rating of 5 was given to whiteness index because the color is the first attribute that is evaluated by the consumers and hence a very important criterion.

Also, since the objective of developing the product is to become an alternative souring agent, the TA was also given a rating of 5. Other factors were given 3 because it was also desired to create a product that is health beneficial (i.e., antioxidant activity, total phenolics), water soluble (i.e., water solubility index), and less spacious (i.e., bulk density). The selection of a fit model (i.e., linear, quadratic, cubic, 2FI) for each response, followed the suggestion of the software where R^2 was as high as possible. The model became significant when the p -value was less than 0.05, otherwise, not significant (i.e., pH) for p -value greater than 0.10. The lack-of-fit test was also performed for the residuals such that the lack of fit was not significant when $p < 0.05$, otherwise, the lack of fit

Table 3 Statistical analysis ($p \leq 0.05$) of responses for optimization of *batuan* fruit powder

Response	Goal	Importance	Fit Model	Two-factor ANOVA		Lack-of-fit Test
				R^2	p -value	p -value
BD (g / mL)	Maximize	3	Quadratic	0.8029	< 0.0001 s	< 0.0001 s
pH	NA	NA	Quadratic	0.2750	0.2055 ns	0.5956 ns
TA(g citric acid / 100 g)	Maximize	5	Quadratic	0.7174	< 0.0001 s	0.5874 ns
TSS (°Bx)	Maximize	3	Quadratic	0.7344	< 0.0001 s	0.0021 s
WI	Maximize	5	Linear	0.6219	< 0.0001 s	0.2443 ns
Antioxidant activity (% DPPH inhibition)	Maximize	3	2FI	0.6516	< 0.0001 s	0.0020 s
Total phenolics(mg GAE / g sample)	Maximize	3	Quadratic	0.5277	0.0049 s	0.0037 s
WAI(g sediment / g)	Maximize	3	Quadratic	0.6552	0.0002 s	0.0018 s
WSI(g dissolved solids / 100 g)	Maximize	3	Quadratic	0.8906	< 0.0001 s	0.0134 s

NA: not applicable, GAE: gallic acid equivalent, 2FI: two-factor interaction, s: significant, ns: not significant

Table 4 Predicted and observed properties of optimized *batuan* fruit powder (50.0 °C drying temperature, 106 ppm SMS, $D = 0.578$)

Physicochemical / Functional Property	Predicted	Actual Value	Error (%)
Whiteness index	46.48	48.30 ± 1.51	3.77
Total phenolics(mg GAE / g sample)	15.70	31.13 ± 0.90	49.58
Antioxidant activity(% DPPH-scavenging activity)	27.14	26.04 ± 0.80	4.21
Titrateable acidity(g citric acid / 100 g sample)	23.86	26.56 ± 0.61	10.15
Total soluble solids (% Brix)	4.67	4.9 ± 0.1	4.71
pH	NA	1.58 ± 0.07	NA
Water absorption index(g sediment / g sample)	3.3662	3.0094 ± 0.1148	11.86
Water solubility index(g solids / g sample)	20.572	21.837 ± 5.647	5.79
Bulk density (g / mL)	0.655	0.661 ± 0.001	0.91

NA: not applicable, GAE: gallic acid equivalent

was significant for $p > 0.10$. For a response to be considered in the optimization, the model should be significant while the lack of fit should be not significant. In the study, only the TA and WI satisfied the criteria; the two responses were also given the highest importance (rating of 5).

However, the other responses were still included in the optimization even if the lack of fit was significant because the models were still “useful” and these responses, in a practical sense, were important considerations in the production of the *batuan* fruit powder. Consequently, the lack of fit of some of the models in the optimization process may compromise the accuracy of the obtained optimum condition. Nevertheless, the responses with significant lack of fit were given less importance (rating of 3) compared to TA and WI.

Thus, the optimum temperature and SMS concentration were calculated as 50.0 °C and 106 ppm, respectively. Also, values of responses corresponding to the optimum condition were predicted by the software (Table 4). However, the desirability was found to be 0.578 which is relatively low.

The overall desirability of 0.578 was obtained as the geometric mean of all the low individual desirability values, each response having an either high or low individual desirability. Since the optimization of SMS concentration and drying temperature involved 8 “significant” responses, then the resulting optimum value, together with the desirability, would lie midway to satisfy all the responses (Kuhn, 2012).

4. Adoption of predicted optimum conditions for *batuan* fruit powder

The computed optimum value was followed to produce optimized *batuan* fruit powder. The drying of *batuan* pulp (containing 106 ppm SMS) took 48 h at 50 °C to produce the brittle dried pulp ready for grinding.

(1) Physicochemical and functional properties of optimized *batuan* fruit powder

After the production of the optimized product, the powder was tested for its physicochemical and functional properties to verify the predicted values as shown in Table 4.

Table 4 reveals that some of the observed values agree with the predicted values, such as whiteness index, antioxidant activity, TSS, WSI, and bulk density, with low percent error. On the other hand, high percentage of error was observed in values for total phenolics. This may be a result of lack of fitness of its predictive model (as seen in Table 3) and desirability that gave more importance to WI and TA. For each response, an appropriate model (i.e., linear, quadratic, cubic, 2FI) which was also suggested by the software, was needed to be established to describe its variation with SMS concentration and drying temperature. However, based on statistical analysis at 5 % level of significance, except for WI and TA, the lack of fitness of most of the responses were found to be significant. Even so, the models were still found to be useful since they were significant based on statistical analysis and were necessary for optimization. Nonetheless, the actual values of most of the responses (i.e., WI, total phenolics, TA, WSI, BD) were found to be better than the predicted values.

(2) Sensory evaluation of optimized *batuan* fruit powder

Sensory evaluation is the ultimate test for product development. Through Quality Scoring, the panelists were able to evaluate the sensory properties of the optimized sample. Table 5 shows the scores given by the panelists.

Based on Table 5, the powder had very light brown color, perceivable weak aroma, and perceivable weak off-odor. The powder was also considered acceptable based on general acceptability.

IV Conclusion

Optimization of processing condition for *batuan* fruit powder was performed. The concentration of SMS in the wet pulp

Table 5 Mean scores of sensory attributes of optimized *batuan* fruit powder (50.0 °C drying temperature, 106 ppm SMS, $D = 0.578$)

Sensory Attribute	Value
Color	1.15 ± 0.59
Aroma	4.84 ± 0.01
Off-odor	3.33 ± 0.39
General acceptability	10.44 ± 0.08

The mean values were obtained from duplicate experiment runs and $n = 10$ semi-trained panelists.

Range of scores are based on 15-cm scale.

Color: 0: very light brown , 5: light brown , 10: medium brown , 15: dark brown

Aroma: 0: weak, 15: strong

Off-odor: 0: not perceivable, 15: perceivable

General acceptability: 0: not acceptable, 5: slightly acceptable, 10: acceptable, 15: very acceptable

was varied at 0, 125 and 250 ppm, while the drying temperature was set at 50, 60 and 70 °C. Complete factorial design was employed to determine the 9 treatments. RSM and desirability function were applied such that there were 2 factors (drying temperature and SMS concentration) and 8 “significant” responses based on Two-factor ANOVA. Regression models were generated from the responses. The models were utilized to calculate the optimum drying temperature and SMS concentration (50.0 °C and 106 ppm, respectively) as well as the overall desirability value, $D = 0.578$. The optimum conditions were adopted to produce the powder, and the powder was analyzed of its physicochemical, functional, and sensory properties. Most of the predicted values of physicochemical and functional properties of the optimized powder were close to the actual values as shown by low percent errors. Based on sensory evaluation by Quality Scoring, the product was found to be acceptable.

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