Production of spray-dried Sarawak pineapple *(Ananas comosus)* powder from enzyme liquefied puree

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Abstract

Spray drying is used widely for converting liquid food products into powder form as the dried powder is known to have a longer shelf life at ambient temperature, convenience to use and low transportation expenditure. In this study, the Sarawak pineapple puree was spray-dried and the characterization of the resulting powder was performed. The process of enzyme liquefaction was optimized with Pectinex® Ultra SP-L and Celluclast® 1.5 L (single and combined treatment) at different concentrations (0–2.5 %) and incubation time (0-2.5 hours). The combined treatment with both enzymes (1.5% v/w Pectinex® Ultra SP-L + 0.5% v/w Celluclast® 1.5 L, 1.5 hour) was found to be the best parameter, which produced purees with the lowest viscosity of 67.98 ± 4.27 cp. Optimization of spray drying process was carried out using different inlet temperatures (150-180°C) and maltodextrin concentrations (15-30 % w/w). Results indicated that the spray-dried powder produced at 160°C with 15% w/w of maltodextrin has the highest yield (31.63 %). The spray-dried powder was further characterized for the moisture content ($6.00 \pm 0.63\%$), water activity (0.36 ± 0.01 A_w), hygroscopicity ($17.35 \pm 0.64\%$), bulk density (0.46 ± 0.04 g/cm³) and solubility (87.33 ± 2.08 seconds). The fruit powder of this study can be incorporated into different fruit added –value products, such as fruit juice, yogurt, jelly and other beverages.

Introduction

Malaysia is well-known for its large diversity of tropical fruits. Although the demand for fresh consumption of tropical fruits is increasing, the high sugar and moisture contents in tropical fruits make them highly perishable, resulting in a short shelf life and limiting their availability throughout the year (APO, 2006). Furthermore, insufficient storage spaces and processing facilities available further contribute to the deterioration and wastage of tropical fruits during peak harvesting season (APO, 2006). Thus, preservation of these excessive tropical fruits into valuable product such as fruit powder has become increasingly popular in recent years.

Dehydration is a process involving the removal of moisture content from a food product. Fresh fruit can be transformed into dry particulates using different dehydration methods, such as freeze drying, solar drying, vacuum drying, oven drying and spray drying (Bala *et al.*, 2003; Gabas *et al.*, 2007; Jittanit *et al.*, 2010; Botha *et al.*, 2012). Spray drying is a common drying method which involves spraying finely atomized solution droplets into a chamber where hot and dry air rapidly evaporates the solvent and converts the droplets into dry particulates (Jaya

sundera *et al.*, 2010). The resulting spray-dried powder is of good quality, low water activity, easier transportation and storage (Tonon *et al.*, 2009). Spray drying is also known to be extremely useful for the encapsulation of heat sensitive food ingredients such as polyphenols that are found commonly in tropical fruits (Fang and Bhandari, 2011).

Nevertheless, there are hurdles during spray drying such as stickiness of powder and its handling due to the natural characteristic of the fruits (Chegini and Ghobadian, 2007). Such scenario is especially common for fruit juice with high sugar content like pineapple, resulting in low product yield and operational problems (Tonon *et al.*, 2008). Therefore, drying agent is used to prevent the depostion of powder onto the drying chamber wall. Maltodextrin is a widely used drying agent that are able to reduce the stickiness of the powder by encapsulating the juice particles (Ng *et al.*, 2012).

The characteristics of the feed used, such as the viscosity and solids concentration, are essential parameters in spray drying (Tonon *et al.*, 2008). This is to ensure that the feed is pump into the spray dryer effortlessly and an acceptable amount of yield will be obtained (Grabowski *et al.*, 2006). Studies have shown that macerating enzymes are able to hydrolyze

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the soluble cell wall components in fruit juice pulp such as pectin, cellulose and hemicelluloses (Ambroziak, 2010). The enzyme liquefaction of the puree can lower the viscosity, facilitate easy pressing, increase juice yield and improve quality in terms of clarity, filterability, flavour and colour (Godfrey *et al*., 1996; Galante *et al*., 1998; Uhlig, 1998; Kashyap *et al*., 2001; Tochi *et al*., 2009).

Sarawak pineapple *(Ananas comosus)* is a local clone of the Smooth Cayenne cultivar found in Peninsular Malaysia. It is popular due to its vigorous growing capabilities and bears large fruit of about 2.5 kg which is relatively larger compared to other cultivars. Its flesh is pale yellow, very juicy with fairly high acid of 0.5-0.9% and sugar content of 12-16° Brix. Research on the preservation of Sarawak pineapple powder by spray drying are yet to be established. There is limited scientific information on how encapsulating agent and drying conditions may influence the physicochemical properties of the spray-dried Sarawak pineapple powder. Thus, the objectives of this study were: a) to optimise the parameters of enzyme liquefaction in terms of enzyme concentrations and incubation time for the production of Sarawak pineapple puree; b) to evaluate the optimum spray drying parameters by using different inlet temperatures and percentage of maltodextrin concentrations in the production of Sarawak pineapple powder; and c) to characterise the physicochemical properties of spray-dried Sarawak pineapple powder produced.

Materials and Methods

Materials

Sarawak pineapple *(Ananas comosus)* used in this study was purchased from Econsave Alam Damai which is located at Cheras, Kuala Lumpur. Maltodextrin (DE 10-12) was obtained from Bronson and Jacobs, Australia.

Pre-treatment process and Sarawak pineapple puree production

The fresh Sarawak pineapples *(Ananas comosus)* were cleaned and washed. The crown, skin and eyes of the pineapple were removed. Small slices (50 mm X 50 mm X 50 mm) of the pineapple were blended into the puree using the Waring® blender (National, Malaysia) at the maximum speed for 30 seconds. The puree was kept at 4ºC until further processing.

Enzyme liquefaction

Single and combined enzyme treatments

For liquefaction, 300 g of homogenised pineapple puree was individually treated with different concentrations (0-2.5% v/w, at an interval of 0.5% v/w) of Pectinex® Ultra SP-L (Novozymes, Denmark). The enzyme-added purees were incubated at 50°C and 100 rpm. The TSS, viscosity and pH of the enzyme liquefied puree were measured at an interval of 30 minutes for 2.5 hours. The enzyme in the treated puree was deactivated at 90ºC. The same procedures were repeated by using Celluclast® 1.5 L (Novozymes, Denmark). Combined enzymes treatment was then performed by adding the optimised Pectinex® Ultra SP-L concentration with Celluclast® 1.5 L at different concentrations (0-2.5% v/w). The same steps were repeated for the combination between optimum concentrations of Celluclast® 1.5 L obtained from single enzyme treatment with different concentrations of Pectinex® Ultra SP-L (0-2.5% v/w). Viscosity was chosen as the key evaluation used to determine the best parameter for enzyme liquefaction. Liquefied puree with the lowest viscosity was favourable as it will maximize the amount of juice yield and prevent the clogging of spray dryer (Phisut 2012).

Spray drying

Optimisation of inlet temperature and maltodextrin concentration

The best enzyme concentration and incubation time determined was used to produce the feed for spray drying. The aqueous extract of pineapple puree was mixed with 30% w/w maltodextrin at a ratio of 1:1. The aspirator rate, pump rate, air flow rotameter and the pneumatic nozzle cleaner speed of the spray dryer (Model B-290, Büchi, Switzerland) were kept constant at 100%, 20%, 40 mm and 6, respectively. The spray drying process was first optimized using different inlet temperatures from 150-180°C at an interval of 10°C. The procedure was then repeated by using different concentrations of maltodextrin, 15-30% w/w (interval of 5% w/w) at the fixed optimised inlet temperature.

The powders obtained from the collecting vessel after each spray drying process were immediately weighed and vacuum packed. The mass of the spray-dried powder which showed the highest yield was chosen as the optimised powder. The process yield was calculated based on the relationship between the total soluble solid (TSS) in the resulting powder and the TSS in the feed mixture (Tonon *et al*., 2008).

All the powders produced were then subjected to physicochemical analyses.

Physicochemical analyses

Total soluble solids (TSS), pH, moisture content and water activity

The TSS contents of the enzyme liquefied purees were measured by a portable hand refractometer (Atago®, Japan) with a scale of 0-32°Brix. The pH value was measured using a digital pH meter (Jenway, model 3505, UK) and the moisture content of the powders were determined based on AOAC (1984) method. The water activity of the samples was measured by using a water activity meter (Novasina, Labmaster, Switzerland) at $25\pm1.0°C$.

Viscosity

The viscosities of the enzyme liquefied purees were measured by a viscometer (Brookfield, Model DV–II+ Pro).

Colour measurement

The colour of the samples was measured using Hunter Laboratory Colorimeter (Model SN 7877, Ultra-scan, Hunter Associates Laboratory, Virginia).

Hygroscopicity

Hygroscopicity was determined according to the method of Cai and Corke (2000), with slight modifications. 2 g of spray-dried powder produced were placed in a glass desiccator at $25\pm1.0°C$ containing saturated Na_2SO_4 solution (81% RH). After 1 week, the samples were weighed. The hygroscopic moisture (hygroscopicity) was expressed as g of moisture per 100 g dry solids (g/100g).

Bulk density

Bulk density of the samples was determined based on Goula and Adamopoulos (2005) method with slight modifications. A total of 5 g of Sarawak pineapple powder were added into 100 ml graduated cylinder. The cylinder was then tapped mechanically by using a rubber hose until non-significant volume change was observed. The ratio between the mass of the powder and the volume occupied in the cylinder determines the bulk density value.

Solubility

The solubility of the spray-dried powders was analyzed based on Phoungchandang and Sertwasana (2010) method with slight modifications. 1 g of sample powder was dissolved in 300 ml of distilled water which was heated on a hot plate to maintain the temperature at 70°C. The mixture was stirred by using a magnetic stirrer at speed '6'. The time used to dissolve each of the sample powder completely was recorded.

Reconstitution of Sarawak pineapple powder

The spray-dried powder which obtained the highest yield was chosen for the reconstitution study. The powder was rehydrated to the same TSS content as the initial TSS of the liquefied Sarawak pineapple puree, TSS ± 0.5 (Youssefi *et al.*, 2009). 4 g of powder was weighed and dissolved in 100 ml of warm water. The TSS of the solution was checked and compared to that of the optimised puree. More powder was added into the solution until the initial TSS content was achieved. The pH and colour analysis were also done on the reconstituted powder.

Statistical analysis

All experiments were conducted in triplicates (n=3) and an analysis of variance (ANOVA) was performed. Statistical analyses were performed using IBM SPSS software (IBM SPSS Statistics 19). Data were statistically analyzed using one-way ANOVA and Tukey tests. The data were expressed as mean ± standard deviations. The confidence interval at 95% ($p < 0.05$) was used for statistical significance.

Results and Discussion

Enzyme liquefaction

Viscosity was significantly reduced from an initial of 214.97±7.821cp after enzyme treatment. Viscosity is useful to identify the optimum enzyme liquefaction parameter because low viscosity can maximize the amount of juice yield and prevent the clogging of spray dryer (Phisut, 2012). From Table 1, combined enzymes (1.5% Pectinex® Ultra SP-L + 0.5% Celluclast® 1.5 L) produced purees with the lowest viscosity (67.98±4.27 cp) at 1.5 hour. The decrease in viscosity was due to the synergistic effects of pectolytic and cellulolytic enzymes (Schweiggert *et al.*, 2008). The degradation of pectin and cellulose fibrils by both enzymes led to the reduction in water holding capacity, thus, releasing the free water into the system. The presence of unbound water is responsible for the overall reduction in viscosity (Sin *et al.*, 2006; Norjana and Noor Aziah, 2012).

Spray drying

Optimisation of inlet temperature

The results tabulated in Table 2 shows that inlet temperature can significantly affect the yield of

Table 1. Optimised conditions for each liquefied
pineapple purees treated with single
and combined enzyme(s)

Enzyme concentrations	Incubation time (hr)	Viscosity (cp)
1.5% $^v/_w$ Celluclast® 1.5 L	1.5	126.46±4.433
1.5% $^v/_w$ Pectinex® Ultra SP-L	2.0	103.46±2.627
1.5% $^v/_w$ Celluclast® 1.5 L + 1.0% $^v/_w$ Pectinex® Ultra SP-L	1.5	93.15±2.907
1.5% $^v/_w$ Pectinex® Ultra SP-L + 0.5% $^v/_w$ Celluclast® 1.5 L	1.5	67.98±4.265

powder. Specifically, the yield obtained at 160°C (27.60±0.719 %) was higher compared to that at 150°C (24.51±0.738 %), possibly due to the greater efficiency of heat and mass transfer processes when high inlet temperature was applied (Cai and Corke, 2000). A gradual decrease in yield was then observed at 170°C (20.98±2.586 %) and 180°C (16.05±1.159 %). According to Leon-Martinez *et al.* (2010), melting of powder and cohesion of powder on the chamber wall of spray dryer at higher temperature can result in lower powder yield. The inlet temperature of 160°C was selected as the optimised parameter for spray drying of Sarawak pineapple puree.

A significant decrease in the moisture content and bulk density of the spray-dried pineapple powder was observed at the inlet temperature of 180°C (Table 3). Higher inlet temperature leads to rapid heat transfer between the product, drying air and water evaporation (Chegini and Ghobadian, 2005; Phisut, 2012), resulting in the reduction of moisture content. The moisture contents of the pineapple powders from the present study were between 3-5%, which are similar to those dried tea powder (Sinija *et al.*, 2007). Moisture content below 10% is adequate to ensure that the food powder produced is microbiologically safe (Ng *et al.*, 2012). Increase in inlet temperature also has a greater tendency of producing porous powder (and thus reduced bulk density) as the total volume occupied by the particles increases (Walton, 2002).

Optimisation of maltodextrin concentration

Table 4 showed a decreased in the powder yield (%) as the concentration of maltodextrin increased. Nevertheless, insufficient carrier agent may produce sticky powder (Phisut, 2012). Thus, optimum maltodextrin concentration is essential to ensure the production of high-yield and non-sticky powder. The present study showed that spray drying with 15% w/w maltodextrin has the highest powder yield (31.63±2.179 %).

Table 2. The yield of Sarawak pineapple powder spray-dried under different inlet temperatures (150-180°C) at fixed 30% w/w maltodextrin

Inlet temperature (°C)	Powder yield (%)
150	24.51±0.738[c]
160	27.60±0.719[a]
170	20.98±2.586[b]
180	16.05±1.159[d]

Data were expressed in mean ± standard deviation based on triplicate readings (n=3)
[a-c] Values in each row with different superscripts are significantly different (p<0.05)

Table 4. The yield of Sarawak pineapple powder spray-dried under different maltodextrin concentrations (15-30% w/w) at fixed 160°C

Maltodextrin concentration (% w/w)	Powder yield (%)
15	31.63±2.179[a]
20	28.93±1.949[ab]
25	27.62±0.564[ab]
30	25.69±1.860[b]

Data were expressed in mean ± standard deviation based on triplicate readings (n=3)
[a-b] Values in each column with different superscripts are significantly different (p<0.05)

As seen from Table 5, the water activity was ranged from 0.17-0.36 A_w, which are considered to be microbiologically safe (Quek *et al.*, 2007; Perez and Schmalko, 2009). Reduction of hygroscopicity was detected as the maltodextrin concentration increased. Maltodextrin has low hygroscopicity in nature; therefore, the addition of maltodextrin into the mixture will lower the overall hygroscopicity level of the product (Phisut, 2012). Maltodextrin also has superior water solubility (Phisut, 2012), an attribute which made maltodextrin a popular carrier agent in spray drying (Cano-Chauca *et al.*, 2005). Shorter time was needed to dissolve the spray-dried powders as the concentration of maltodextrin increased.

The colorimetric results (Table 5) indicated that the L* values increased proportionally with the amount of maltodextrin added. This shows that the spray-dried powders appeared very light and white in colour as more maltodextrin were added (HunterLab, 2008). However, a* values decreased slightly as the concentration of maltodextrin increased. The negative a* values indicating that the spray-dried powders were very light green in colour . The colour changes of spray-dried guava and watermelon powders due to addition of maltodextrin were also noted by Chopda and Barrett (2001) and Quek *et al.* (2007), respectively.

Reconstitution of optimised powder

The pH of the reconstituted powder was at 4.533

Table 3. Physicochemical analysis of Sarawak pineapple powder spray-dried under different inlet temperatures (150-180°C) at fixed 30% w/w maltodextrin

Analysis	Inlet temperature (°C)			
	150	160	170	180
Moisture content (%)	4.47±0.833[a]	4.00±0.100[a]	3.27±1.026[a]	2.93±0.702[b]
Water activity (A_w)	0.213±0.006[a]	0.238±0.052[a]	0.162±0.021[a]	0.181±0.042[a]
Hygroscopicity (%)	17.39±1.334[a]	18.11±2.245[a]	18.90±0.779[a]	19.37±2.479[a]
Bulk density (g/cm^3)	0.485±0.026[a]	0.477±0.225[a]	0.436±0.019[ab]	0.391±0.044[b]
Solubility (sec)	65.00±2.65[a]	63.67±3.48[a]	59.00±1.00[a]	60.00±4.58[a]
Colour				
L*	96.144±0.112[a]	96.053±0.230[a]	96.203±0.294[a]	96.301±0.481[a]
a*	-0.461±0.064[a]	-0.446±0.018[a]	-0.523±0.027[a]	-0.388±0.141[a]
b*	4.005±0.103[a]	4.552±0.560[a]	4.255±0.168[a]	4.652±0.767[a]

Data were expressed in mean ± standard deviation based on triplicate readings (n=3)
[a-b] Values in each row with different superscripts are significantly different (p<0.05)

Table 5. Physicochemical characteristics of Sarawak pineapple powder spray-dried under different concentration of maltodextrins (15-30% w/w) at fixed 160°C

Analysis	Concentration of maltodextrin (% w/w)			
	15	20	25	30
Moisture content (%)	6.00±0.630[a]	5.16±0.821[ab]	4.19±0.605[b]	4.23±0.252[b]
Water activity (A_w)	0.357±0.008[a]	0.247±0.046[b]	0.190±0.004[bc]	0.172±0.007[c]
Hygroscopicity (%)	17.35±0.640[a]	15.07±0.723[b]	14.36±0.735[bc]	13.38±0.409[c]
Bulk density (g/cm^3)	0.457±0.042[a]	0.373±0.035[ab]	0.331±0.046[b]	0.328±0.025[b]
Solubility (sec)	87.33±2.08[a]	72.33±2.52[b]	70.67±2.08[bc]	64.00±3.61[c]
Colour				
L*	95.039±0.603[b]	95.168±0.102[ab]	95.653±0.307[ab]	95.967±0.147[a]
a*	-0.067±0.034[a]	-0.146±0.009[b]	-0.179±0.018[b]	-0.255±0.026[c]
b*	5.294±0.843[a]	5.219±0.126[a]	4.585±0.508[a]	4.986±0.332[a]

Data were expressed in mean ± standard deviation based on triplicate readings (n=3)
[a-c] Values in each row with different superscripts are significantly different (p<0.05)

± 0.021, which was higher than pH 3.813 ± 0.032 of the puree. Overall, the colour of the reconstituted powder (L*= 87.445 ± 0.001; a*=--2.559 ± 0.012; b*=-5.447 ± 0.016) was lighter than the puree (L*= 55.280 ± 1.664; a*=-1. 594 ± 0.020; b*=28.209 ± 0.399).

Conclusions

Combined treatment of Pectinex® Ultra SP-L and Celluclast® 1.5 L at 50°C for 1.5 hr was the best enzyme liquefaction parameters, which produced puree with the lowest viscosity of 67.98 ± 4.27 cp. Spray-dried powder produced under the inlet temperature of 160°C and 15 % w/w of maltodextrin showed the highest yield of powder, 31.63 %. Physiochemical characteristics of the optimised spray-dried powder were successfully determined. Reconstituted juice was found to be lighter in colour and had a higher pH value (4.53 ± 0.02) than its initial pH (3.81 ± 0.03). The product of this study has the potential to be developed into natural additives with added value and longer shelf-life.

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