



Original Research Article

Effect of drying on total phenolic compounds, antioxidant activities and physical properties of palm sugar

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ABSTRACT

This research aimed to study the effect of drying on total phenolic compounds, antioxidant activities and physical properties of palm sugar. The vacuum drying was operated at the temperature of 40, 50 and 60 °C for 3, 4 and 5 h. It was found that vacuum drying at 60 °C for 5 h gave the lowest moisture content of 0.98% (wet basis). The highest phenolic content of palm sugar dried at 40°C for 3 h was 16.29 mg of gallic acid equivalents/mg (dry weight). The drying condition of palm sugar at low temperature yielded higher phenolic content than that of high temperature. The antioxidant activity of dried palm sugar determined by 2, 2 - diphenyl-1- picrylhydrazyl (DPPH) scavenging activity and ferric reducing antioxidant power (FRAP) showed the highest value of 59.34% and 1.11 μmol/g of sample at the drying of 40°C for 3 h. These values were positively correlated to total phenolic compounds. The dried palm sugar had lowest water activity (a_w) and solubility time of 0.31 and 31.66 sec when operated at the drying of 60 °C for 5 h. Based on the results of these studies, the drying condition of palm sugar at 40°C for 3 h delivered the highest total phenolic content and antioxidant activities of dried palm sugar.

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INTRODUCTION

The Asian Palmyra palm (*Borassus flabellifer* Linn.) is the source for palm sugar and originated in tropical countries such as Sri Lanka, India, Myanmar, Indonesia, Malaysia and Thailand (Naknean *et al.*, 2010). In Thailand, most population of palmyra palms are found in southern part including Phetchaburi province. This is an important tree in that all parts of plant can be used by humans living in the local area. Production of palm sugar in Thailand is approximately one hundred thousand tons per year (Department of Phetchaburi Agricultural Extension, 2013). Palm sugar is directly consumed or used as an ingredient for making desserts, cake, food coating or drinks (Ho *et al.*, 2008). Traditionally, palm sugar is collected from palm sap. Typically, a few pieces of payorm wood (*Shorea roxburghii* G Don) are added to the palm sap during collection as a natural preservative. The sap is boiled in an opened pan evaporator for a few hours until it becomes concentrated (~80 °Brix). The production of concentrated palm sugar has increased due to its convenience and longer shelf life. Apart from the sweetness of palm sugar, it may have antioxidant activity from the phenolic compounds releasing from pieces of payorm wood. However, no information on antioxidant activities of palm sugar is available in the literature. An antioxidant can prevent the oxidation of other molecules by quenching reactive free radicals. Damage mediated by free radicals can result in the disruption of membranes. This has been considered to be linked with many chronic health problems such as cancers, inflammation, aging and atherosclerosis (Kinsella *et al.*, 1993). Therefore, the antioxidant function could directly impact on health-promoting effects in the prevention of degenerative diseases (Kim *et al.*, 2000).

In the present study, vacuum drying was applied to produce dried palm sugar. The influences of temperature and time on the physicochemical qualities of the final dried palm sugar products were investigated. The changes in the total phenolic compounds and antioxidant activities were also determined.

MATERIALS AND METHODS

Preparation of palm sugar for sap of juice, concentrated and vacuum dried forms

Palm sap of juice was collected after cutting inflorescences at the site of harvesting in Tha Yang, Phetchaburi province, Thailand. The container used for collecting juice was sanitized with hot water and let dry at room temperature. Chip of payorm wood (3 g) was added to the container and palm juice was harvested in the morning after 12 h of collection. After that the palm sap of juice was filtered through the cheese cloth and kept in an icebox and then transported to the Department of Agro-Industrial, Food and Environmental Technology, Bangkok. Palm sap of juice was kept frozen at -18 ± 2 °C until analysis. For making the concentrated form, palm juice was concentrated by conventional stirring and heating at approximately 105°C for 3 h. The concentrated palm sugar having total soluble solids of 80 °Brix were kept in sealed polyethylene bag at 5 ± 3 °C in a refrigerator for 1 month. The concentrated palm sugar was dried by using a vacuum oven (Vacucell 22, Germany) at 40, 50 and 60 °C for 3, 4 and 5 h. The dried sample were ground and sieved through a 10 mesh. The samples were kept at room temperature in sealed aluminium foil bag until used.

Determination of physicochemical properties

The vacuum-dried powders was analyzed for the color on the CIE (Commission Internationale de L'Eclairage) indicating L^* , a^* , b^* by a colorimeter (Hunter Lab, Colorquest, USA). Hue angle measures the property of the color and it is calculated according to the Equation 1.

$$\text{hue} = \tan^{-1} (b^* / a^*) \quad (1)$$

The moisture content was determined according to the method of AOAC (2000). Measurement of water activity was determined by a water activity meter (Aqua Lab CX-2, USA). Solubility was determined using a modification of the method of Quek *et al.* (2007). Briefly, 50 mg of sample was placed in 50 mL of distilled water. The mixture was mixed by using a vortex mixer (Vortex genie 2, USA) at half speed. The time (sec) to complete solution was recorded using an electronic timer.

Total phenolic compounds

Total phenolic compounds were determined by the method of Al-Farsi *et al.* (2005). Gallic acid was used as a standard. Briefly, 200 mL of sample solutions were mixed with 1500 µl of Folin - Ciocalteu reagent and left it stand for 5 min. After that, 1500 µl sodium carbonate was added, the reaction was allowed to stand for 90 min. The absorbance was measured at 725 nm. The results were expressed as mg of gallic acid per 100 g of dried sample.

Antioxidant activity of dried palm sugars

DPPH radical scavenging capacity and ferric reducing antioxidant power (FRAP) were used to determine antioxidant activity. The free radical DPPH scavenging capacity was determined followed by the method of Mansouri *et al.* (2005). 500 mL of sample were added to 1500 µl of 0.1 mM DPPH radical solution. The mixture was left for 30 min in the dark at 25 ± 2 °C. The DPPH radical was determined by measuring the absorbance at 517 nm and calculated the percentage of DPPH radical inhibition according to Equation 2.

$$\% \text{ DPPH inhibition} = [(A_c - A_s) / A_c] \times 100 \quad (2)$$

where A_s is the absorbance of sample solution, A_c is the absorbance of the DPPH solution

The other determination of antioxidant activity was investigated by using the ferric reducing antioxidant power assay (Buasod, 2006). 500 µl of the samples were added to 1.25 mL of phosphate buffer and 1.25 mL of potassium ferricyanide (1%). The mixtures were incubated at 50°C for 20 min and then 1.25 mL of trichloroacetic acid solution (10 %) was added. Then, the mixture was combined with 1.25 mL of deionized water and 0.25 mL of FeCl_3 (1%). The ferric-tripyridyltriazine (Fe^{3+} -TPTZ) complex having an intense blue color was measured at 595 nm. Results were reported as µmol of gallic acid per g of sample.

Statistical analysis

Each experiment, from sample preparation to analysis, was performed in triplicate. The results were expressed as means \pm standard deviation. The analysis of variance (ANOVA) was used to compare the mean values at $P < 0.05$. Mean values and pooled standard error of the mean were then calculated.

RESULTS AND DISCUSSION

Determination of physicochemical properties

Generally, the palm sap of juice and concentrated palm sugar are light brown and brown color. The color of palm juice showed lightness (L^*), redness (a^*) and yellowness (b^*) of 26.97, -0.48 and 7.95, respectively. Concentrated palm sugar had $L^* = 33.85$, $a^* = 29.15$ and $b^* = 57.12$. Heating during the concentration process changed the color to an intense brown color due to caramelization and Maillard reactions. The form of palm sugar also changed from liquid to semisolid. Pigment of payorm wood contributed to the color of palm juice. The palm juice with no payorm wood will be fermented during long time of harvesting. In practice, the juice containers were collected and pooled after 8-12 h. However, most concentrated palm sugars were produced from small scale enterprises or local farmers which do not have a standard method for concentration. The different times and temperatures used for concentration cause changes in color. Naknean (2010) reported that the palm sugar in Songkhla province had L^* , a^* and b^* values between 61.49 to 87.53, 1.46 to 3.52 and 12.41 to 19.31, respectively. Location differences and process factors (time and temperature) lead to the changes in palm sugar color. For vacuum dried palm sugar, when different factors of temperature and time were applied, the color of dried palm sugar showed slight differences in $L^*a^*b^*$ (Table 1). The L^* values tended to be increase with increasing time of drying but the yellowness decreased. However, all dried palm sugars showed a green-yellow shade color as indicating from the Hue angle values. During drying process, the a^* value increased with increasing temperature and time. Naknean *et al.* (2009) reported that during the heating process of concentrating and drying, increasing in a^* value may be a result of non-enzymatic browning, including Maillard and caramelization reactions.

Table 1 Physical properties of vacuum dried palm sugar

Temp (°C)	Times (h)	L^*	a^*	b^*	Solubility times (sec)	Moisture content (%)	a_w
40	3	70.08 ± 0.06 ⁱ	1.05 ± 0.00 ^g	25.28 ± 0.05 ^a	53.16 ± 3.18 ^e	2.32 ± 0.05 ^a	0.46 ± 0.01 ^e
	4	73.41 ± 0.08 ^f	2.45 ± 0.02 ^d	21.09 ± 0.05 ^e	57.23 ± 1.57 ^f	2.21 ± 0.01 ^{ab}	0.46 ± 0.01 ^e
	5	75.99 ± 0.10 ^d	2.72 ± 0.02 ^{ab}	18.07 ± 0.05 ^g	57.66 ± 1.53 ^f	2.47 ± 0.01 ^{ab}	0.41 ± 0.00 ^d
50	3	71.30 ± 0.03 ^h	0.98 ± 0.02 ⁱ	23.04 ± 0.02 ^c	40.86 ± 0.98 ^d	2.23 ± 0.08 ^{ab}	0.42 ± 0.00 ^d
	4	79.47 ± 0.01 ^c	1.93 ± 0.03 ^e	18.54 ± 0.01 ^f	43.20 ± 2.00 ^d	2.11 ± 0.22 ^{bc}	0.36 ± 0.01 ^b
	5	82.46 ± 0.24 ^a	2.69 ± 0.01 ^b	14.96 ± 0.10 ⁱ	41.66 ± 1.00 ^d	1.10 ± 0.17 ^{de}	0.35 ± 0.00 ^b
60	3	72.89 ± 0.07 ^g	1.64 ± 0.05 ^g	22.30 ± 0.15 ^d	37.83 ± 0.58 ^c	1.91 ± 0.03 ^c	0.38 ± 0.00 ^c
	4	75.05 ± 0.10 ^e	2.57 ± 0.02 ^c	23.82 ± 0.04 ^b	34.70 ± 0.52 ^b	1.35 ± 0.19 ^d	0.31 ± 0.01 ^a
	5	80.66 ± 0.56 ^b	2.75 ± 0.03 ^a	17.57 ± 0.13 ^h	31.66 ± 1.22 ^a	0.98 ± 0.24 ^f	0.31 ± 0.00 ^a

All data are the mean ± SD of three replications.

The different superscripts in the same column denote the significantly different ($P < 0.05$).

The moisture contents of juice and concentrated palm sugar were 80.65% and 35.68%. When the vacuum drying was applied, the moisture content of the dried powders decreased with the increased temperature and time (Table 1). A higher temperature, the rate of heat transfer to the particles is greater. The driving force for moisture evaporation increased, thereby reducing the water content or moisture of dried sample (Kelly *et al.*, 2014). Concentrated sugar dried at 60 °C for 5 h had moisture content of 0.98%.

Water activity (a_w) is a physical property that has a direct impact on microbiological property and shelf life of food. From a safety point of view, no microorganisms grow if the a_w value is lower than 0.5 (Barbosa-Canovas *et al.*, 2007). Water activities of the dried palm sugar powders (Table 1) were in the range of 0.31-0.41. However, the water activities of dried palm sugar slightly increased with decreased drying time and temperature. Therefore, the vacuum-dried palm sugar powders produced were relatively stable on the microbiological standpoint.

Solubility of palm sugars was determined by measuring the time (sec) to completely dissolve in water. The palm sugar dried at 60°C for 5 h had the greatest solubility (lowest solubility time). Vacuum dried sugar processed at lower temperatures were less soluble. The lower temperature of vacuum drying could cause the slower evaporation rate, thereby producing powders with higher moisture content (Quek *et al.*, 2007). The dried samples had a high tendency ofglomeration or forming a hard sheet lay on the top of water surface. This could prevent water molecules from diffusing through the particle (Chegeni and Ghobadian, 2005).

Total phenolic compounds

Payorm wood is typically used as a natural preservative that has an impact on microorganisms. Increasing environmental temperatures during harvesting favors the rapid growth of microorganisms that enhance spoilage. Palm sap of juice had pH of 4.19- 5.23. Therefore, the cleanliness is very important during harvesting step. The microorganisms found in palm sugar juice are *Saccharomyces cerevisiae* and lactic acid bacteria (Chanthachum and Beuchat, 1997). The payorm wood can delay spoilage in juice by reducing microbial populations (Charoenchai *et al.*, 2003). Tannin is the most important

phenolic compound found in bark of payorm (*Shorea roxburghii* G Don) (Chanthachum and Beuchat, 1997). The total phenolics of stem bark of payorm in acetone and methanol extracts were 65.74 ± 8.70 and 67.67 ± 4.90 mg/mL, respectively (Subramanian *et al.*, 2013). The total phenolic content of palm sugars which derived from pieces of payorm wood was measured. The total phenolic content of palm juice was 156.85 ± 0.35 mg of gallic acid equivalents per 100 g (dry weight basis). The phenolic content of concentrated palm sugar had 40.06 ± 0.85 mg of gallic acid equivalents per 100 g (dry weight basis). The

phenolic contents of vacuum dried palm sugars had ranged from 2.14 to 16.29 mg of gallic acid equivalents per 100 g (dry weight basis) (Table 2). The total phenolic contents of dried palm sugar decreased with the increased temperature of drying. The influence of different drying processes on the concentration of phenolic compounds can be attributed to the varying stability of different phenolic compounds under the drying conditions (Joshi *et al.*, 2011). The results suggested that the loss of phenolic compound may be from the vacuum drying process. Based on the benefit of phenolic compounds, many properties of antioxidative, antimicrobial, anticarcinogenic, antimutagenic, antiallergic and antiinflammatory activities were reported (Kim *et al.*, 2003; Pereira *et al.*, 2013).

Using the FRAP method, there was a significant difference between the temperature of 40, 50 and 60 °C, while at the drying of 60 °C demonstrating the lowest values. The lowest FRAP value of drying at 60 °C for 5 h was 0.66 µmol per g. The highest FRAP value was 1.11 µmol per g at drying of 40 °C for 3 h. The reducing power increased with increasing the phenolic content of sample (Subramanian *et al.*, 2013). These results suggested that palm sugar may donate electrons to reactive free radicals, converting them into more stable non-reactive species and terminating the free radical chain reaction (Lobo *et al.*, 2010).

Table 2 Total phenolic compounds and antioxidant activity (DPPH and FRAP method) content in palm sugars.

Temp (°C)	Times (h)	Total phenolic compounds (mg of gallic acid equivalents/100 g)	DPPH radical scavenging (%)	FRAP value (µmol/g)
40	3	16.29± 0.53 ^a	59.34± 0.42 ^a	1.11± 0.050 ^a
	4	12.73 ± 0.52 ^b	47.09 ± 5.18 ^d	1.06 ± 0.006 ^b
	5	12.27 ± 0.34 ^b	48.81 ± 2.62 ^{cd}	1.04 ± 0.004 ^b
50	3	12.27 ± 0.01 ^b	52.62 ± 0.66 ^b	1.06 ± 0.025 ^b
	4	9.42 ± 0.17 ^c	49.44 ± 0.39 ^{bcd}	1.08 ± 0.010 ^{ab}
	5	7.28 ± 1.08 ^d	48.24 ± 0.94 ^{cd}	1.07 ± 0.014 ^b
60	3	4.04 ± 0.58 ^e	51.62 ± 0.85 ^{bc}	0.98 ± 0.003 ^c
	4	2.91 ± 0.23 ^f	45.83 ± 0.19 ^{de}	0.85 ± 0.016 ^d
	5	2.14 ± 0.19 ^f	43.33 ± 0.63 ^e	0.66 ± 0.034 ^e

All data are the mean ± SD of three replications.

The different superscripts in the same column denote the significantly different ($p \leq 0.05$).

Antioxidant activity of dried palm sugars

In general, the antioxidant activity are derived from phenolic compounds and/or vitamins (Kim *et al.*, 2003). Palm sugars were shown to have phenolic content which can act as an antioxidant. The potency of antioxidant property could be affected by the parameters of drying process. In this investigation, the widely used DPPH test and FRAP assays have been applied. The dried palm sugar sample with highest content of total phenolics of 16.29 mg of gallic acid equivalents per 100 g (dry weight) had the highest radical scavenging value (59.43 %) (Table 2). The statistical analysis showed that temperature and time of drying had significant effect on the antioxidant activity of dried palm sugar. The DPPH scavenging capacity showed that the increased drying temperature decreased the radical scavenging value. Increased total phenolic content may enhance the action of radical scavenging (Pinela *et al.*, 2012). Piga *et al.* (2003) found that different drying processes affect the antioxidant activity. The increased antioxidant activity could be explained by the greater antioxidant power from the hydroxyl group of polyphenols that can be quench the DPPH radical at the intermediate stages of oxidation or the formation of new compounds with higher antioxidant activity.

In the reducing power assay, the presence of antioxidants in the dried palm sugar results in the reduction of Fe^{3+} /ferricyanide complex. The reducing power of compound may serve as a significant indicator of its potential antioxidant activity reported by Meir *et al.* (1995).

CONCLUSION

Vacuum drying can be used to produce dried palm sugar. Increasing temperature and time altered the physicochemical properties of the vacuum-dried powders. Based on the results of these studies, the drying condition of palm sugar at 40 °C for 3 h delivered the highest total phenolic content and antioxidant activities of dried palm sugar. Vacuum drying can cause losses of phenolic compounds and antioxidant properties.

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