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Effect of Drying Temperature on Malaysia Pomelo (*Citrus grandis* (L.) Osbeck) Pomace Residue under Vacuum Condition

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ABSTRACT

Pomelo pomace (PP) from Malaysia Tambun White (PO52) variety, dried at different temperature under vacuum condition were evaluated. PP was obtained after extraction process and contained high moisture content (MC). Dried PP can be used as an alternative to livestock feed, hence, dried concentrated form was preferable. Nevertheless, drying process significantly affects the physico-chemical properties of a dried product. Therefore, the present study was carried out to discover the effects of drying temperature on the physico-chemical properties (MC, pH, vitamin C, browning index (BI) and total color change) of PP. The PP was dried at different drying temperature (50, 60, 70, 80 and 90°C) using a vacuum drying (VD) oven for 24 hr. The freeze drying (FD) process was used as a control. The result showed the MC gradually decreased with increasing drying temperature, of which similar trend was also observed for the water activity and total soluble solids (TSS). In contrast, pH showed increment in value with elevated drying temperature. Interestingly, at 90°C, vitamin C of VD (30.38 mg / 100 g DW) was higher than FD pomelo pomace (21.10 mg / 100 g DW). BI significantly increased as temperature increased. However, no significant changes were observed for total color change after VD. In conclusion, VD at 90°C showed the most recommended temperature because the composition of quality properties at this

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temperature was not significantly varied with control. These fundamentals information of pomelo pomace could be the guideline for postharvest technologist and food processing manufacturers for various applications.

Keywords: Malaysian tambun pomelo, pomelo pomace, quality, temperature, vacuum drying

INTRODUCTION

Pomelo (*Citrus grandis* (L.) Osbeck) also known in Malay as 'Limau Bali, Limau Abung, Limau Tambun, Limau Besar and Limau Betawi', a native citrus fruit from South East Asia (Morton, 1987).

A significant quantity of the pomelo residues is generated in both Thailand and Malaysia every year. This is because of high export demand and domestic consumption of the pomelo fruits. Juice production from citrus fruits including pomelo leads to a higher yield production of pomelo pomace (Bai et al., 2013).

Pomelo pomace is the pulpy residues obtained after the juice has being extracted. The yield of pomelo pomace (16%) was recorded and collected from the whole pomelo pulp. Interestingly, previous research had revealed that pomelo residues containing bioactive compounds (phenolic compounds) were capable to promote health benefits (Chang & Azrina, 2017; Shah et al., 2012; Toh et al., 2013) and it also could become a potential source of dietary fibre due to high recovery from pomelo albedo (Zain et al., 2014). Dried citrus pulps were discovered as potential alternative sources of cereal substitute in livestock feed which contained high net energy particularly for dairy cow (Wadhwa & Bakshi, 2013). In addition, Bhatnagar et al. (2015) reviewed the potential source of production on renewable, low cost and sustainable adsorbent for water treatment from pomelo waste.

Pomelo pomace (PP) was obtained during a post-extraction process, whereby it might retain the remaining juice that contributed to higher moisture content and total soluble solids. Microorganism reaction favors this condition and leads to spoilage of fresh pomelo residues. Therefore, drying process reduces the probability of spoilage occurrence due to the removal of free moisture available for microorganism reaction in the dried concentrated form of pomelo pomace. Therefore, selection of drying condition is essential to prolong the shelf-life while retaining the quality (water activity, pH, total soluble solids (TSS), vitamin C and color analysis) of dried pomelo pomace.

Vacuum drying is generally applied for preservation of the heat-sensitive composition by reducing the pressure that leads to evaporation of water molecules (Karam et al., 2016). As a result, it allows limited changes to physicochemical properties and preserving ascorbic acid (Oikonomopoulou & Krokida, 2013).

Recently, researchers have studied the quality of juice from pomelo and its peels. For instance, chemical composition and physical properties (length, width, thickness, surface area, projected areas and volumes) of the juice and the whole pomelo fruits were discovered for Ledang (PO55) and Tambun (PO52) varieties (Buang et al., 2015; Shah et al., 2012). As drying process occurs, the physicochemical properties of citrus fruit would change substantially. According to Shamsudin et al. (2015a), conventional drying method shows an effect on the color of pomelo pulp waste after being dried at 50, 60 and 70°C. Nevertheless, limited studies providing detailed of vacuum drying on physicochemical properties of pomelo fruits (pomelo pomace) have been conducted. Therefore, the aim of this research is to determine the effects of different drying temperature on physicochemical properties of Malaysia's pomelo pomace.

MATERIALS AND METHODS

Plant Materials

The pomelo (*Citrus grandis* (L.) Osbeck) was obtained from P052 Tambun White variety, Perak in Malaysia. Then, the flesh was inserted into a fruit press juicer (CL-003AP, Interglobal International, LTD, Taiwan) and manually operated. The juice and pomelo pomace (waste) were separated and pomelo pomace was used as raw material for the drying process.

Drying Process

Freeze drying process was conducted by weighing pomelo pomace (~100 g) and frozen in an ultra-low freezer (MDF-U2086S; Sanyo, Japan,) at -85°C overnight and lyophilized using freeze dryer at at $4.5 \times 10^{-2} \sim -9.9 \times 10^{-3}$ mmHg (VirTis Benchtop K, PA, USA) for 96 hr. During vacuum drying (VD), the pomelo pomace was weighed (~60g) and was spread as a thin layer with a 02cm thickness on an aluminum foil (21 cm x 21 cm) and dried in vacuum oven (VD23, Binder GmbH, Germany) with pressure 90 – 110 mbar at 50, 60, 70, 80 and 90°C. The samples were dried for 24 hr, and stored at -20°C until the physicochemical analysis was conducted.

Physicochemical Properties

Determination of Moisture Content. The moisture content of pomelo pomace was conducted according to AOAC method 925.10 (AOAC, 2000). The initial moisture content of the fresh pomelo pomace was 82.46% fresh weight.

Determination of Water Activity. Water activity (aw) was determined using Aqualab series 3TE (Decagon Devices Inc., Pullman, WA, US). The water activity was measured with an Aqualab water activity meter (Aqualab series 3TE, Decagon Devices Inc., Pullman, WA, USA) with a dew point hygrometer at 25°C.

Determination of Total Soluble Solids (TSS). The total soluble solids (TSS) content was expressed in °Brix, performing the measurement in a pomelo pomace using refractometer (Atago CO Ltda, Itabashi-Ku, Tokyo, Japan). Distilled water was used as a blank prior to analysis of the sample.

Determination of pH. The pH was recorded using pH meter (Fisher Scientific Accumet, AP 72 Waterproof Hand-held Meters, Hampton, New Hampshire, United States) at room temperature.

Determination of Ascorbic Acid. Ascorbic acid (Vitamin C) content of pomelo pomace was determined based on 2, 6-dichloroindophenol titrimetric method (AOAC, 2000) as it is a simple and rapid method to determine Vitamin C content.

Determination of Color. Furthermore, color analysis (L, a, b, hue, and chroma) including browning index (BI) and total color change (ΔE) were performed using a color reader (CR-10, Konica Minolta, Japan.) following Rahman et al. (2016) method. The lightness (L*), redness/greenness (a*) and yellowness/blueness (b*) was evaluated. These parameters were used to calculate the hue angle, chroma, BI and total color changes. The hue angle (*h*), chroma (C*), BI and total color changes (ΔE) was calculated using Equation 1, 2, 3, and 4 respectively. As total color changes involve the differences between dried product color with the fresh product, the parameter of fresh color of pomelo pomace involved are L (53.01), a (3.54) and b (24.87).

$$h^\circ = arctan\left(\frac{b*}{a*}\right) \quad (1)$$

$$Chroma = \sqrt{a*^2 + b*^2} \quad (2)$$

$$BI = \frac{[100(x - 0.31)]}{0.17} \quad (3)$$

where $x = \frac{(a+1.75L)}{5.645L+a-3.012b}$

$$\Delta E = \sqrt{(L* - L_o)^2 + (a* - a_o)^2 + (b* - b_o)^2} \quad (4)$$

Statistical Analysis

A Duncan's test was performed using IBM SPSS Statistics 21.0 to differentiate and evaluate the significance between the mean values. The confidence limits was considered as 95% ($p < 0.05$). The values were stated as the mean ± standard deviation.

RESULTS AND DISCUSSIONS

Table 1 shows the physicochemical properties consisting of moisture content, water activity, and TSS of dried pomelo pomace at different drying temperatures. FD pomelo pomace showed higher MC than a recommendable level of moisture content (Table 1). Despite the samples being completely frozen, the ice crystals formed (Rahman et al., 2016; Singh & Heldman, 2001) during frozen state might not completely be removed that leading to higher levels of moisture content. After that, sublimation process (evaporation of solid to vapor phase) occurs in a vacuum operation (Dzung, 2012). As can be seen, the moisture content of vacuum dried (VD) pomelo pomace was gradually decreased as temperature increased. This highlights the condition of the reduction of water vapor concentration at the product surface due to vapor pressure gradient between the product and the environment (partial vacuum in the drying chamber) (Dev & Raghavan, 2012; Karam et al., 2016). As suggested by Geankoplis (2003) moisture content (MC) less than 10% is preferable to make microorganism inactive in a dormant state preventing the undesired changes from occurring. Therefore, vacuum drying at 70, 80, and 90°C of pomelo pomace was less than 10% MC (8.67, 7.31 and 6.46 respectively).

Table 1
Physicochemical properties of dried pomelo pomace at a different drying temperature

Treatment	Temperature (°C)	Moisture content (%)	Water activity (a_w)	TSS (°Brix)
FD (control)		13.95±1.03c*	0.36±0.002c	2.97±0.06a
	50	22.31±0.23a	0.43±0.005a	2.33±0.06b
	60	14.93±0.09b	0.39±0.01b	2.33±0.06b
VD	70	8.67±0.24d	0.38±0.01b	2.17±0.06c
	80	7.31±0.26e	0.38±0.01b	2.03±0.12d
	90	6.46±0.07f	0.38±0.01b	2.23±0.06bc

*Source: Rahman et al. (2016)
Different letters in the same column showed significant difference at (p<0.05). FD: Freeze drying; VD: Vacuum drying.
Note: The value was measured in dry based weight (moisture content g /100 g of dried weight based).

Water activity (a_w) of VD at 50°C showed slightly higher value than FD. It could be due to the presence of bound moisture in the dried pomelo pomace as the pomace is considered a hygroscopic type. However, it was considered lower than recommended water activity level (<0.7) (Geankoplis, 2003) which is safe for the final dried product. Nevertheless, there is no significant (p>0.05) value observed when temperature increased. In contrast, the water activity of the fresh pomelo peels reported by Looyrach et al. (2015) was higher (~0.98) than the present study. Higher a_w value liable to all microbial growth and thus reducing the shelf stability of the fresh material which is not recommended.

Total soluble solids of the VD pomelo pomace (2.33-2.03°Brix) showed a significant reduction (p<0.05) with FD pomelo pomace (2.97°Brix). The reducing value could be due to exposure to heat treatment eventually leading to an oxidative breakdown of the complex material such as organic acid, starch, and sugars to simple molecules such as carbon dioxide and water (Yau et al., 2010).

The pH of VD pomelo pomace (4.65-4.95) was lower than FD (6.07) pomelo pomace (Figure 1). Low value of pH in VD indicates a higher concentration of acidity (H+ ion) in comparison with FD. It could be due to dissociation of carboxylic acid during heat treatment from the vacuum drying process. The lower pH value might be due to the dissociation of carboxyl group in amino acids from protein in pomelo pomace (Shamsudin et al., 2015b).

As for vitamin C in Figure 1, during elevated temperature, vitamin C of VD pomelo pomace increased simultaneously (11.54-30.83 mg AA/100 g DW), particularly for VD90 which was higher than freeze dried (FD) (21.10 mg AA/ 100 g DW). It could be due to the reduction of moisture content leading to increment of the aqueous phase viscosity and reactants precipitation affecting the diffusion and retaining the vitamin C (Santos & Silva, 2008). In addition, this effect could be attributed to the oxygen-free environment, which promoted a protective atmosphere and reduced the aerobic degradation of ascorbic acid preventing the oxidation process (Kongsoontornkijkul et al., 2007; Wang et al., 2018). Thomkapanich et al. (2007) observed similar phenomenon during low-pressure superheated steam drying (LPSSD) of Indian gooseberry at 75°C.

Color is an essential quality index of foods and agricultural products. The unstable color changes lead to the low marketing value and reduce the quality. The color, browning index and total color change of dried pomelo pomace at different drying temperature are

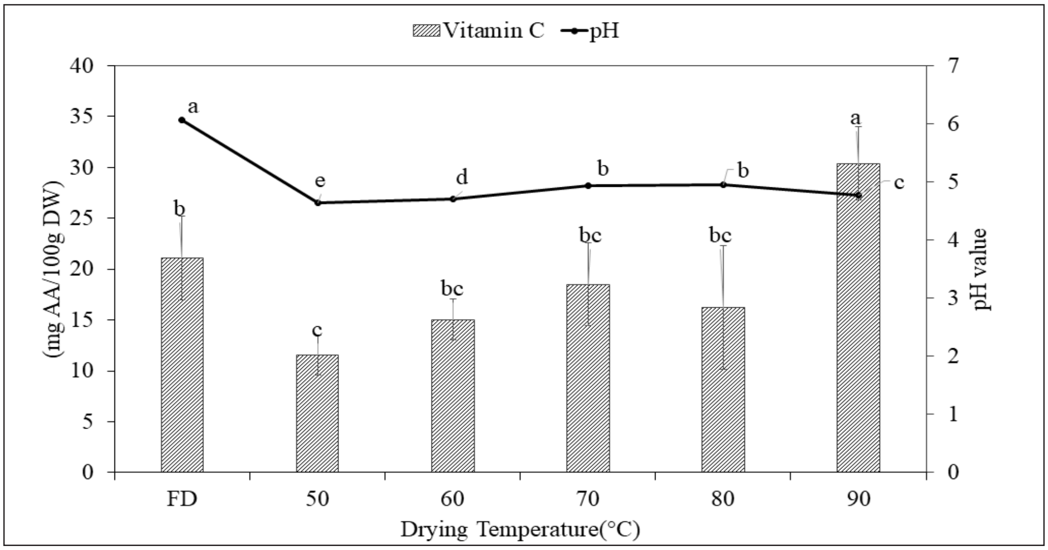

Figure 1. Effect of different drying temperature on pH and vitamin C of pomelo pomace (PP)

shown in Table 2 and Figure 2 respectively. Color denoted by CIE lightness (L, a, b) and browning index (BI) of vacuum drying process were changed significantly ($p<0.05$). Similar changes were observed in hue angle and chroma parameter. During elevated temperature, the hue angle reduced significantly ($p<0.05$). It might indicate the hue angle was in the range < 90 that corresponds to yellow-orange-red (brown) color. The lower the hue angle value indicates the darker color of the material. Meanwhile, chroma represents the intensity of the color. Table 2 shows that the chroma level increases significantly (p<0.05) during elevated drying temperature. It could be due to the instability and degradation of the color

Table 2
Color of dried pomelo pomace (PP) at different drying temperature

Treatment	Temperature (°C)	L	a	b	hue	Chroma
FD (control)*		66.50±0.17a	6.87±0.61d	28.00±0.35b	86.55±0.53a	28.83±0.47c
	50	43.60±0.70c	6.63±0.20d	28.26±0.91b	86.84±0.21a	29.03±0.90c
	60	45.37±0.42b	7.63±0.10c	30.83±0.10a	86.50±0.07a	31.76±0.12b
VD	70	44.87±0.25b	8.36±0.06b	31.43±0.36a	85.95±0.07b	32.52±0.36ab
	80	45.20±0.20b	8.56±0.15ab	31.76±0.50a	85.84±0.15b	32.90±0.50a
	90	43.50±0.66c	9.03±0.26a	30.56±0.15a	85.01±0.34c	31.87±0.09b

*Source: Rahman et al. (2016)
Different letters in the same column showed significant different at ($p<0.05$). FD: Freeze drying; VD: Vacuum drying

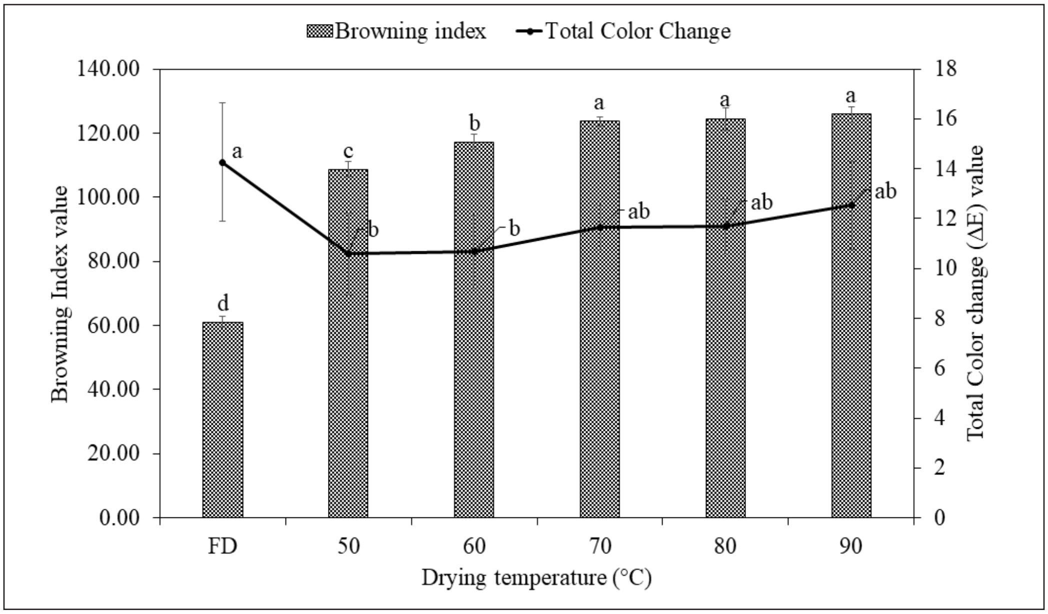

Figure 2. Effect of different drying temperature on the browning index and total color change of pomelo pomace (PP)

pigment when dried at a higher temperature *(*Chong et al., 2008; Lee et al., 2012). In addition, acceleration of Maillard and enzymatic reactions could be the reason of brown pigment formation *(*Adam et al., 2000; Kiranoudis et al., 1993) or caramelization process occurred during the increment of drying temperature (Díaz et al., 2003; Ghanem et al., 2012). A similar trend has been discovered by dried lemon slices using pulsed vacuum drying process during elevated different drying temperature (60, 65, 70 and 75°C) (Wang et al., 2018). Nevertheless, in general, no significant changes were observed for total color change particularly VD pomelo pomace at 70, 80 and 90°C compared to FD pomelo pomace.

CONCLUSION

In brief, vacuum drying at 90°C showed the most recommended drying temperature in vacuum condition. Vitamin C is the essential micro vitamin which can be an indicator of the product. By taking these compound into consideration, VD 90 is selected as the best condition as it comprises a higher value of retention. The composition of physico-chemicals and color properties at this temperature were equivalent or better than that of freeze drying process. Identification of the best drying operation applied for preservation method is highly recommended. Minimum changes of physico-chemical of the final product after drying process is highly preferable. Therefore, different drying operation is suggested to achieve the requirement of final product (natural supplement for vitamin C).

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