Effects of Post-Drying Methods on Pomelo Fruit Peels

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Abstract Changes in physicochemical properties of moisture, ash, and fat contents, and color, due to freeze and oven post-drying treatments on flavedo, albedo, and lamella pomelo fruit peels were investigated. Physicochemical properties influence consumer acceptability and only a few studies are known. Pomelo peels were subjected to freeze drying and conventional drying at 60°C. Fresh pomelo peel was used as a control. Post-drying treatment changes in moisture, ash, and fat contents were observed, compared to controls. Minimal color changes were observed for freeze drying, compared with oven drying for flavedo, albedo, and lamella. Useful information for evaluation of drying treatments that can be used on pomelo peels is provided.

Keywords: post-drying, freeze-dried, oven-dried, pomelo peel

Introduction

Pomelos (*Citrus grandis* (L.) Osbeck) are large citrus fruits of Family Rutaceae that are native to southeastern Asia, including Malaysia (1) where popular pomelo varieties are Tambun, either white or pink, Shatian, and Melomas. Toh *et al.* (2) reported that white Tambun is of interest due to juiciness, sweetness, and a delicious taste. Pomelos are usually consumed as fresh fruits or subjected to juice extraction while peels are discarded (3) accounting for a by-product amount of approximately 30% of the initial pomelo weight. Previous research has found that citrus fruit peels are a promising source of dietary fiber and comprise several functional compounds that are beneficial to health (4-6). Di Scala and Crapiste (7) reported that in order to preserve bioactive compounds and prolong the peel shelf life, a dehydration process is important to remove water for minimization of microbial spoilage and deterioration reactions. Moreover, moisture removal disrupts many moisture-mediated deteriorative reactions and retards growth and reproduction of microorganisms and, eventually, leads to changes in physicochemical attributes, including crust formation, shape, texture, and chemical changes, and loss of nutritional value and aroma (7,8). Dehydration methods commonly applied in fruit and vegetable preservation are hot air, solar, microwave, foam-mat, spray, spouted bed, freeze drying, and osmotic dehydration. The most popular method applied is convective drying due to low cost and ease of handling (9); however, quality degradation can occur (10). Freeze drying is the most effective method for retaining product quality, but is expensive. However, the

principle behind both drying methods is material volume and water content reduction for inhibition of biochemical enzyme and microbial reactions. Other advantages of drying include safe waste storage, reduction of the degree of hazard, and reduced transportation costs (11).

Dehydration techniques effect quality properties of texture, color, taste, and nutritional values of fat and ash contents. Although drying is an important process for preservation of fruit residues, little information is available regarding development of color and physicochemical changes in pomelos post-drying, which refers to removal of water from solids as a vapor after harvest that causes product quality deterioration in nutritional composition and color and, consequently, affects customer first impressions regarding fruit quality. It has been known that (10,12), in order to fulfill a demand for high-quality shelf-stable dried products, selection of a drying method for production of a high quality final product is important. This information would be useful for post-harvest handling, and for breeders, gardeners, importers, and exporters. Therefore, this study was carried out to analyze changes in physicochemical properties of moisture, ash, fat contents, and color of post-oven and freeze drying treatments of dried pomelo fruit peels, including the flavedo, albedo, and lamella.

Materials and Methods

Materials Tambun White (*Citrus grandis*) PO52, the most cultivated and popular pomelo variety among consumers in Malaysia, was used

in this study. Fruits harvested from a commercial orchard in Tambun, Perak, Malaysia in April of 2015 were selected based on uniformity of size, color, and commercial maturity stage. Fruits were washed to remove soil and contaminants, then manually peeled and separated from pulp. Peels were divided into 3 different parts known as flavedo, a greenish peel color, albedo, a whitish spongy peel, and lamella, the skin that covers the pulp. Peel parts were then cut cubes for albedo of approximately 1×1×1 cm and slabs for flavedo and lamella of 1.0–2.0±0.2 mm thickness before application of drying treatments. In addition, this study did not involve any experimentation with human or animal subjects performed by any of the authors.

Drying experiments The 2 methods of dehydration used were convection oven drying using a DO6836 oven (Mettler GmbH, Schwabach, Germany) and freeze drying using a Benchtop K device (SP Scientific, Gardiner, NY, USA).

Oven drying: Peels were subjected to drying treatments at 60°C for 24 h in a DO6836 oven (Mettler GmbH) and dried peel samples were stored at –18°C in a freezer (HVF-301S; Hesstar Corporation Sdn. Bhd., Shah Alam, Malaysia) prior to subsequent proximate analysis.

Freeze drying: During freeze drying, peel samples were frozen at –80°C in an ultra low temperature freezer (MDF-U2086S; Sanyo Electric Co., Ltd., Osaka, Japan) and freeze dried for 96 h at 4.5×10^{–2}–9.9×10^{–3} mmHg (Benchtop K; SP Scientific) until completely dry. Evaporation occurred when the product temperature reached optimal conditions of 0.008 mmHg for 13.82 h, resulting in conversion of water to the vapor phase under vacuum (13). Dried peel samples were stored at –18°C in a HVF-301S freezer (Hesstar Corporation Sdn. Bhd.) prior to further analysis.

Physicochemical analysis

Determination of moisture content: The moisture content was determined following the Association of Official Analytical Chemists (AOAC) Method 925.10 (14). In brief, 2 g of a peel sample was put into an aluminum dish and placed in an oven (DO6836; Mettler GmbH) at 105°C overnight until a constant weight was achieved. The moisture content was determined using Eq. 1, which represents moisture loss in weight of dried peel samples:

$$\frac{\left(\frac{\text{Initial weight of filled}}{\text{aluminum dish}}\right) - \left(\frac{\text{Final weight of filled}}{\text{aluminum dish}}\right)}{\left(\frac{\text{Initial weight of filled}}{\text{aluminum dish}}\right) - \left(\frac{\text{Initial weight of empty}}{\text{aluminum dish}}\right)} \times 100 \quad (1)$$

Determination of ash content: The ash content was determined using furnace incineration following an AOAC method 900.02 (15). In brief, 1 g of pomelo peel was placed in a crucible and incinerated at 550°C for 6 h in a KSL-1700X ashing muffle furnace (MTI Corporation, Richmond, CA, USA) until ash was obtained, followed by cooling in a desiccator and weighing using an analytical balance (XSE 204; Mettler Toledo, Greifensee, Switzerland). The percentage ash content

was obtained using (Eq. 2):

$$\frac{(\text{Weight of ash})}{(\text{Weight of sample})} \times 100 = \% \text{Ash} \quad (2)$$

Determination of fat content: The fat content was determined using the Soxhlet extraction method following an AOAC method 960.39 (16) with a Soxtec extraction apparatus (SoxtecTM 2050; Foss Analytical, Edenprairie, MN, USA) (17) with adjustment of temperature and time and attachment of thimble containing 1 g of pomelo peel. After extraction, extracted material in a flask was dried at 105°C in an oven (OF-G22W; Jeio Tech, Daejeon, Korea) for 1 h, then placed in desiccator. The fat content was obtained using Eq. 3:

$$\frac{(\text{Weight of fat})}{(\text{Weight of sample})} \times 100 = \% \text{fat} \quad (3)$$

Color analysis: The pomelo peel color was determined using a CR-10 colorimeter (KonicaMinolta, Tokyo, Japan) (18) as lightness (L*) within a range of 0 (black) to 100 (white). Chroma (C*) indicates the purity of color with reference to red-green (+/–a*) and yellow-blue (+/–b*) color components. Similar color components were used for determination of hue angle (h*), which indicate how the human eye sees the color (19). C* and h* values were calculated using Eq. 4 and 5, respectively. The browning index (BI), which indicates the purity of the brown color of peel samples, was used as a reference for browning color changes (20). The BI value was calculated using Eq. 6 while total color change (E) was calculated using Eq. 7, where the subscript “0” refers to the color reading of fresh pomelo peel. Fresh flavedo, albedo, and lamella were used as references and a minimal E change denoted minimal color change from reference material.

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (4)$$

$$h^* = \arctan\left(\frac{b^*}{a^*}\right) \quad (5)$$

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

$$\text{where } x = \frac{(a^* + 1.75L)}{(5.645L + a^* + 3.012b)} \quad (6)$$

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (7)$$

Results and Discussion

Physico-chemical composition

Effects of post-drying on moisture content: Effects on moisture content during post-drying of pomelo peels using oven and freeze drying treatments were evaluated and changes are shown in Table 1. Fresh pomelo peel was used as a control. Abirami *et al.* (21) showed that removal of moisture is necessary for preservation of peel quality and shelf life extension of products while preventing microbial deterioration and enzyme activities. The moisture content of fresh

Table 1. Moisture content (%) of pomelo peels affected by different drying techniques

Sample	Fresh	Freeze dried* ¹⁾	Oven dried*
Flavedo	80.590.47 ^{aA2)}	9.330.53 ^{bA}	5.150.48 ^{cB}
Albedo	77.691.31 ^{aB}	8.630.31 ^{bA}	5.880.27 ^{cAB}
Lamella	78.180.51 ^{aB}	5.670.06 ^{cB}	6.330.28 ^{bA}

¹⁾*Values are means of moisture content on a dry basis (g of water/100 g of dry solid) from triplicate determinations.

²⁾Values denoted with the same small letter within the same row are not significantly ($p>0.05$) different between fresh and dried pomelo peels. Values with the same capital letter within the same column are not significantly ($p>0.05$) different between fruit peels.

flavedo was 80.59% whereas the moisture contents of freeze-dried and oven-dried peels were 9.33 and 5.15%, respectively. There was a significant reduction ($p<0.05$) in moisture content with drying, compared with controls, for both drying methods. The moisture content of dried flavedo was reduced to 9.33% for freeze drying and to 5.15% for oven drying, perhaps due to differences in moisture removal between the 2 methods. Freeze drying involves water evaporation via sublimation, which converts water to the vapor phase in a vacuum at a low temperature (13). In contrast, oven drying involves a high temperature of 60°C that leads to simultaneous water evaporation via heat and mass transfer (10). Freeze-dried flavedo showed a significantly ($p<0.05$) higher moisture content than oven-dried flavedo, perhaps due to ice crystals formed (22) during the freezing phase that were not completely removed during freeze drying. Results for oven-dried flavedo in this study were consistent with previous reports by Abirami *et al.* (21) and Zain *et al.* (3).

Fresh albedo also showed higher moisture content of 77.69% than freeze-dried at 8.63% and oven-dried albedo at 5.88%. Post-drying affects the moisture content of albedo. Oven-dried albedo with a content of 5.88% showed a significantly ($p<0.05$) lower moisture content than freeze-dried albedo at 8.63%, lower values than for pomelo albedo reported by Zain *et al.* (3), and for lemon albedo (23). Thus, oven drying method contributed to rapid removal of water from air filled intercellular spaces of albedo (24), reducing the moisture content.

Post drying of lamella also showed a significant ($p<0.05$) reduction of moisture content, compared with fresh lamella with a content of 78.18%. Fresh lamella possessed a higher moisture content than freeze-dried lamella at 5.67% and oven-dried lamella at 6.33%. A significant ($p<0.05$) reduction in lamella moisture content was observed after freeze and oven drying process, compared with controls. In addition, along with post drying methods, the moisture content of freeze-dried lamella of 5.67% was significantly ($p<0.05$) lower than for oven-dried lamella at 6.33%, perhaps due to application of a vacuum in the freeze drying process and a high vapor pressure differential between the center and the surface of the material that allowed a higher rate of moisture removal from the product than for the oven drying method (9).

There were significant differences ($p<0.05$) in moisture content

Table 2. Ash content (%) of pomelo peels affected by different drying techniques (dry weight basis)

Sample	Fresh	Freeze dried* ¹⁾	Oven dried*
Flavedo	1.830.07 ^{cA2)}	5.920.03 ^{bA}	6.750.21 ^{aA}
Albedo	0.780.01 ^{bb}	2.640.55 ^{aB}	3.600.14 ^{aB}
Lamella	0.870.03 ^{bb}	2.290.57 ^{aB}	3.050.21 ^{aB}

¹⁾*Values are means of ash content on a dry basis (g/100 g of dry solid) from triplicate determinations.

²⁾Values denoted with the same small letter within the same row are not significantly ($p>0.05$) different between fresh and dried pomelo peels. Values with the same capital letter within the same column are not significantly ($p>0.05$) different between fruit peels.

between fresh flavedo at 80.59% and other types of pomelo peels. However, there was no significant ($p>0.05$) difference between fresh lamella at 78.18% and fresh albedo at 77.69%. The higher flavedo moisture content was probably due to an outermost location on the pomelo fruit and a waxy layer covering the surface that acted as a gas barrier to limit evaporation and retain moisture inside the peel (25). Overall, for freeze-dried pomelo peels, 91-94% of the moisture content was reduced whereas the oven drying method reduced the moisture content by 94-95%. Thus, dried pomelo peels can be considered safe for long-term storage (26) with moisture content lower than 13%.

Effects of post-drying on ash content: Post-drying effects on the ash content were evaluated on a dry weight basis (Table 2). The ash content is representative of the total mineral content. In general, fresh pomelo peels showed significantly ($p<0.05$) lower ash contents of 0.78-1.83% than dried pomelo peels at 2.29-6.75%. During the post-drying process, moisture was completely removed and the total peel sample weight consisted of dry material without moisture was used for determination of the ash content (27). In contrast, the total weight of fresh peel samples reflected moisture and material contents. Therefore, the ash content of fresh peel samples was slightly lower than for dried peels. Similar results were reported in previous studies by Abirami *et al.* (21) and Karaman *et al.* (28).

Oven-dried flavedo showed a significantly ($p<0.05$) higher ash content of 6.75% than both freeze-dried at 5.92% and fresh flavedo at 1.83%, perhaps due to the ash content corresponding to the moisture content in oven-dried flavedo, which showed a lower moisture content than freeze-dried flavedo (Table 1). The lower the moisture content of pomelo peels, the higher the ash content. A similar trend was previously reported (19,27), perhaps due to large amounts of inorganic nutrients (28,29) present in flavedo. In addition, the ash content of oven-dried flavedo of 6.75% in this study was comparable to reports for citrus peels of 5.15-6.43% reported by Abirami *et al.* (21).

Oven-dried albedo showed a higher ash content of 3.60% than freeze-dried at 2.64% and fresh albedo at 0.78%. However, the ash content of oven-dried albedo did not differ significantly ($p>0.05$) from freeze-dried albedo, but did differ significantly ($p<0.05$) from fresh albedo. Thus, post-drying of albedo did not affect the ash

content of dried albedo, in contrast with fresh albedo. Nevertheless, results obtained for oven-dried albedo of 3.60% in this study were comparable with the report of Zain *et al.* (3) of 3.41% for dried pomelo albedo, and with the value of 2.94-3.03% for dried orange fiber reported by Crizel *et al.* (30) and Ghanem *et al.* (29).

Oven-dried lamella at 3.05% showed a higher ash content than freeze-dried at 2.29% and fresh lamella at 0.87%. However, the ash content of oven-dried lamella was not significantly ($p>0.05$) different from freeze-dried lamella, but was significantly ($p<0.05$) different from fresh lamella. Thus, the freeze drying and oven drying processes did not affect the ash content of lamella. However, freeze-dried lamella showed lower moisture content than oven-dried lamella (Table 1), but freeze-dried lamella showed a lower ash content than oven-dried lamella, perhaps due to spattering of the freeze-dried lamella during incineration process (27) and eventually minimize the volume and ash content. Only a few studies related to lamella have been reported. Nevertheless, the ash content of oven-dried lamella was in agreement with the pomelo peel fiber value of 3.17-3.82% reported by Abirami *et al.* (21).

The ash content of flavedo differed significantly ($p<0.05$) from albedo and lamella peel types. Flavedo at 1.83-6.75% exhibited a higher ash content, followed by albedo at 0.78-3.6%, and lamella at 0.87-3.05% (Table 2). In addition, the ash content of dried albedo was comparable with the content of dried lamella due to a high mineral content in flavedo, in agreement with citrus peels reported by Abirami *et al.* (21) and Ghanem *et al.* (29). The ash content of pomelo peels can act both as micro and macro elements that provide health-promoting effects in both humans and animals (21).

Effects of post-drying on fat contents: Post drying also affected the flavedo, albedo, and lamella fat content on a dry basis (Table 3). Shamsudin *et al.* (25) stated that the fat content is important for sustaining texture, flavor, and pigments of fruits. Freeze-dried flavedo showed a dry basis fat content of 1.63%, comparable with fresh flavedo at 1.51%. However, fresh and freeze-dried flavedo values were significantly different ($p<0.05$) from oven-dried flavedo at 0.73%, perhaps due to tightly packed parenchymatic cells in flavedo that limited fat loss during freeze drying (30). Thus, oven-dried flavedo exhibited a lower fat content after hot-air drying, which also affected the texture, color, flavor, aroma, and nutritive value (10). In comparison, low temperature freeze-dried flavedo retained a better aroma (28), in agreement with a study reported by Karaman *et al.* (28). Fresh albedo showed a significantly ($p<0.05$) higher fat content of 1.32%, than freeze-dried at 0.60% and oven-dried albedo at 0.13%.

The structure of albedo, which is highly porous and comprised of air-filled intercellular spaces (30), lead to easier loss of essential oils, in agreement with a previous study (31). The fat content of fresh albedo was slightly lower than for oven-dried albedo at 1.56% (3). In contrast, the lamella fat content was not significantly ($p>0.05$) different from freeze-dried albedo and oven-dried flavedo during post-drying. Nevertheless, fat contents were lower than for a

Table 3. Fat content (%) of pomelo peels affected by different drying techniques

Sample	Fresh	Freeze dried* ¹⁾	Oven dried*
Flavedo	1.510.22 ^{aA2)}	1.630.05 ^{aA}	0.730.12 ^{bA}
Albedo	1.320.15 ^{aA}	0.600.05 ^{bB}	0.130.06 ^{cB}
Lamella	0.410.004 ^{aB}	0.750.05 ^{aB}	0.700.30 ^{aA}

¹⁾*Values are means of fat content on a dry basis (g/100 g of dry solid) from triplicate determinations.

²⁾Values denoted with the same small letter within the same row are not significantly ($p>0.05$) different between fresh and dried pomelo peels. Values with the same capital letter within the same column are not significantly ($p>0.05$) different between fruit peels.

previous study reported by Abirami *et al.* (21), which showed a significant difference between dried citrus pulp (5.23-5.45%), dried peel (3.63-4.12%), and dried peel fibers (2.78-3.0%). Fresh flavedo showed higher fat contents of 1.51% than fresh albedo at 1.32% and fresh lamella at 0.41%, perhaps due to the presence of oil glands that are commonly found in stems, leaves, flowers, and fruits located in the outermost part of the rind (31). Generally, oven drying affected the fat content of dried pomelo peels more than the freeze-drying method. Moreover, flavedo possessed higher fat contents than albedo and lamella.

Effects of post-drying on color: Color, an important parameter for quality observation, has been used for determination of the effects of post-drying methods on dried products (10,20). L^* , a^* , b^* , h^* , C^* , BI , and E values of fresh and dried pomelo peels are shown in Fig. 1A for flavedo, Fig. 1B for albedo, and Fig. 1C for lamella. Post-drying significantly ($p<0.05$) affected L^* , a^* , h^* , and BI , and E values of flavedo. The L^* of freeze-dried flavedo of 59.63 showed moderate degradation, compared with fresh flavedo at 61.83. The a^* value of freeze-dried flavedo of -3.30 was similar to the value for fresh flavedo of -7.87. Oven-dried flavedo showed a significantly ($p<0.05$) different BI value of 106.72 than freeze-dried flavedo at 85.07, perhaps due to presence of polyphenoloxidase compounds that reacted with phenolics to form browning pigments (28) during oven drying. However, C^* and b^* values were not significantly ($p>0.05$) different between drying methods. Generally, E values of flavedo were significantly ($p<0.05$) different between oven dried at 14.20 and freeze dried flavedo at 5.40, showing that occurrence of maillard products and enzymatic reactions during the drying process probably lead to a greater degree of total color change for oven-dried flavedo (32).

Albedo color criteria are shown in Fig. 1B. Post-drying significantly ($p<0.05$) affected the color of dried albedo, compared with controls. Oven-dried albedo showed significant ($p<0.05$) differences in L^* , a^* , C^* , b^* , h^* , BI , and E values of albedo, compared with controls. BI values of oven-dried albedo at 78.00 differed significantly ($p<0.05$) from freeze-dried albedo at 45.93, suggesting instability of color pigments in albedo or melanin compounds (brown products) formed during condensation or polymerization of *o*-quinones during drying (33,34). Overall, E values of dried albedo were higher for oven drying

Fig. 1. Color analysis of fresh and dried (A) Flavedo; (B) Albedo; (C) Lamella.

at 16.28 than for freeze drying at 7.64.

Lamella color criteria are shown in Fig. 1C. Dehydration affected the color of dried lamella. Post-drying showed significant ($p < 0.05$) differences in +a, +b, h^* , C^* , BI, and E values, compared with controls. However, there were no significant ($p > 0.05$) differences observed for L^* values between oven-dried at 67.67 and freeze-dried lamella at 69.13. BI values of oven-dried lamella at 76.52 differed significantly ($p < 0.05$) from freeze-dried lamella at 49.97, perhaps due to the presence of heat sensitive compounds, such as proteins and carbohydrates, that change the color of dried fruits (35). Therefore, E

values differed significantly ($p < 0.05$) between oven-dried at 9.53 and freeze-dried lamella at 3.77.

Generally, the freeze drying process resulted in minimal color changes and produced a product quality similar to fresh fruit, compared with oven drying. The color of dried flavedo, albedo, and lamella pomelo peels was significantly ($p < 0.05$) affected by both drying methods, compared with controls.

In conclusion, the freeze drying method resulted in minimal changes in physicochemical properties of ash and fat content, and color, and produced a product quality similar to fresh fruit. Moreover, dried flavedo showed significantly ($p < 0.05$) higher ash and fat contents than albedo and lamella. This study has provided useful information for evaluation of different drying treatments for pomelo peels.

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