PHYSICOCHEMICAL AND ANTIOXIDANT PROPERTIES OF POMELO RESIDUES USING DIFFERENT DRYING METHODS AND KINETIC MODELS OF NARINGIN DEGRADATION

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DOCTOR OF ENGINEERING IN FOOD ENGINEERING

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A DISSERTATION SUBMITTED IN PARTIAL FULFILLMENT
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ชื่อเรื่อง PHYSICOCHEMICAL AND ANTIOXIDANT PROPERTIES OF POMELO RESIDUES USING DIFFERENT DRYING METHODS AND KINETIC MODELS OF NARINGIN DEGRADATION

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บทคัดย่อ

ส้มโอทับทิมบุน (PO52) เป็นส้มพันธุ์พื้นเมืองและเป็นผลไม้ที่ได้รับการยอมรับว่าเป็นผลไม้รสเปรี้ยวที่ใหญ่ที่สุดในโลก และเป็นที่รู้จักกันดีในลิเมาบาหลีประเทศมาเลเซีย ส้มโอนิยมรับประทานเป็นผลสดจึงทำให้มีกากเหลือทิ้งโดยไม่คำนึงถึงประโยชน์ โดยส่วนที่ทิ้งประกอบไปด้วยเปลือก ส่วนเยื่อขาว เส้นใยรอบเนื้อและเยื่อโดยรอบ (~50%) พบว่า กากส้มโอมีปริมาณสารต้านอนุมูลอิสระแตกต่างอย่างมีนัยยะสำคัญ ($p<0.05$) เมื่อเทียบกับผลสด โดยพบสารต้านอนุมูลอิสระหลักในเปลือกส้มโอ คือ นารินจิน ที่มีประโยชน์ด้านส่งเสริมสุขภาพโดยช่วยลดความเสี่ยงในการเป็นโรคหัวใจ แต่อย่างไรก็ตาม กากจากผลส้มโอสดที่มีความชื้นสูง เมื่อทิ้งไว้ในระยะเวลานานจะเกิดการเจริญเติบโตของเชื้อจุลินทรีย์ขึ้นได้ ดังนั้น กระบวนการทำแห้งจึงเป็นวิธีการที่พึงกระทำก่อนเพื่อลดปัญหานี้ การอบแห้งด้วยลมร้อนแบบดั้งเดิม (CD) เป็นกระบวนการทำแห้งที่รู้จักกันดี มีการใช้ลมร้อนเป็นแหล่งให้ความร้อนในการอบแห้งผลิตภัณฑ์ ในขณะที่การอบแห้งแบบสุญญากาศ (VD) เป็นกระบวนการทำแห้งที่จะดึงโมเลกุลน้ำให้ระเหยออกภายใต้สภาวะการลดความดัน นอกจากนั้น ยังไม่มีการศึกษาเกี่ยวกับจลนศาสตร์การอบแห้งของการอบแห้งด้วยลมร้อนแบบดั้งเดิม (CD) กับการอบแห้งแบบสุญญากาศ (VD) ที่ใช้อธิบายพฤติกรรมการอบแห้งของกากเปลือกส้มโอที่อุณหภูมิต่าง ๆ รวมถึงข้อมูลของการเสื่อมถอยของสารนารินจินและความสามารถในการเป็นสารต้านอนุมูลอิสระในกากเปลือกส้มโอในระหว่างการเก็บรักษาค่อนข้างมีอย่างจำกัด ดังนั้น งานวิจัยนี้จึงมีวัตถุประสงค์หลักเพื่อศึกษาผลของการอบแห้งที่อุณหภูมิต่าง ๆ ระหว่าง CD และ VD ที่มีผลต่อคุณภาพของกากเปลือกส้มโอ โดยสภาวะการอบแห้งที่ถูกเลือกให้เหมาะสมจะพิจารณาจากปริมาณสารฟีนอลิกทั้งหมดที่คงเหลือมากที่สุดและการเกิดความสามารถในการต้านอนุมูลอิสระที่สูงสุดโดยแสดงค่าเป็นดัชนี (D^{3} 0.80) ที่วิเคราะห์ได้จากข้อมูลทางสถิติ โดยพิจารณาจากค่าสัมประสิทธิ์การตัดสินใจ (R^2) มากที่สุด และความคลาดเคลื่อนมาตรฐานของการพยากรณ์ (SEE) ต่ำที่สุด นอกจากนี้ มีการศึกษาหาแบบจำลองทางจลนศาสตร์ของการอบแห้งทั้งสองวิธีและสมการการเสื่อมถอยของสารนารินจินในระหว่างการ

เก็บรักษา ผลของสภาวะการเก็บรักษาที่สัปดาห์ที่ 12 อุณหภูมิ 8 องศาเซลเซียส ถูกใช้เพื่อศึกษาลักษณะการเสื่อมถอยของสารนารินจินและความสามารถของการต้านอนุมูลอิสระ ด้วยสมการลำดับศูนย์ หนึ่งและสอง ผลการศึกษาพบว่า การอบแห้งทั้งสองวิธี (CD และ VD) ที่อุณหภูมิต่าง ๆ ส่งผลต่อคุณภาพกากเปลือกส้มโออย่างมีนัยยะสำคัญทางสถิติ ($p < 0.05$) โดยเปลือกส่วนโฟมสีขาวพบสารฟีนอลิกทั้งหมดสูงที่สุดเมื่อเทียบกับส่วนอื่น ๆ ในเปลือกส้มโอเมื่อผ่านการอบแห้งแบบสุญญากาศที่อุณหภูมิ 90 องศาเซลเซียส จึงเป็นสภาวะการอบแห้งที่ดีที่สุดในการอบแห้งส่วนโฟมขาวของเปลือก (D ³ 0.08) แบบจำลองวิธีการแพร่ (Diffusion approach model) นำเสนอผลการทำนายสำหรับแบบ CD ดีที่สุด ขณะที่แบบจำลอง two-term exponential นำเสนอผลการทำนายการอบแห้งโฟมขาวของเปลือกด้วยวิธี VD ดีที่สุด ภายใต้อุณหภูมิการอบแห้ง 50 - 90 องศาเซลเซียส สัมประสิทธิ์การแพร่ประสิทธิผล (D_{eff}) ภายใต้การอบแห้งแบบ CD มีค่าลดลงอย่างเป็นเชิงเส้น ตามอุณภูมิที่ศึกษาโดยมีค่าอยู่ในช่วง 5.00×10^{-7} ถึง 6.98×10^{-7} m^2/s ในขณะที่สัมประสิทธิ์การแพร่ประสิทธิผลการอบแห้งด้วยวิธี VD มีแนวโน้มเพิ่มขึ้นตามช่วงซึ่งอยู่ในช่วง 9.86×10^{-7} - 1.30×10^{-6} m^2/s การหายไปของออกซิเจนพบว่า มีความสัมพันธ์กับอัตราการระเหยความชื้นจากตัวอย่างสู่สิ่งแวดล้อมภายใต้สภาวะสุญญากาศในการอบแห้งแบบสุญญากาศ (VD) อย่างมีนัยยะสำคัญทางสถิติ ($p < 0.05$) นอกจากนี้ สารสกัดหยาบแห้งที่ได้จากการอบแห้งแบบสุญญากาศ (VC) มีปริมาณสารนารินจินมากกว่าที่ได้จากการอบแห้งแบบฟรีซดราย (FC) ในระหว่างการเก็บรักษาที่สัปดาห์เริ่มต้นแตกต่างอย่างมีนัยยะสำคัญทางสถิติ ($p < 0.05$) แต่อย่างไรก็ตาม พบว่า อัตราการเสื่อมถอยของสารนารินจินมีค่าลดลงในสารสกัดหยาบแห้งที่ผ่านการอบแห้งแบบฟรีซดราย (FC) เมื่อเก็บไว้ในระยะเวลาที่นานขึ้น นั่นแสดงให้เห็นว่า สารสกัดหยาบแห้งที่ได้จาก FC มีความคงตัวมากกว่าที่ได้จาก VC นอกจากนี้ลักษณะการเสื่อมถอยของสารนารินจินสามารถอธิบายได้ด้วยสมการลำดับศูนย์ หนึ่ง และสอง โดยพบว่า มีสัมประสิทธิ์การตัดสินใจสูงที่สุดที่ ($R^2 > 0.89$) และให้ค่าความคลาดเคลื่อนมาตรฐานของการพยากรณ์ต่ำที่สุด ($SEE < 0.05$) สุดท้าย งานวิจัยนี้ได้เป็นประโยชน์ในการให้ข้อมูลเชิงลึกเพื่อใช้ในการวิจัยด้านการเก็บเกี่ยว อุตสาหกรรมอาหาร โภชนาการ และอุตสาหกรรมยา ซึ่งจะเป็นข้อมูลเพื่อใช้เป็นทางเลือกใหม่ในการผลิตสารออกฤทธิ์ทางชีวภาพปริมาณสูงจากกากเปลือกส้มโอได้ในอนาคต

คำสำคัญ : ส้มโอ, สารสกัดชีวภาพ, สารต้านอนุมูลอิสระ, แบบจำลองจลนศาสตร์, การอบแห้ง

Title	PHYSICOCHEMICAL AND ANTIOXIDANT PROPERTIES OF POMELO RESIDUES USING DIFFERENT DRYING METHODS AND KINETIC MODELS OF NARINGIN DEGRADATION
Author	Miss Nur Farhana Abd Rahman
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ABSTRACT

Tambun Pomelo (PO52) also known as Limau Bali in Malaysia is a native citrus fruit recognized as the largest citrus fruit in the world. Pomelo is commonly eaten fresh while the pomelo residues (~50%) consist of flavedo, albedo, lamella and pulp waste discarded without acknowledging its potential. A significant amount ($p < 0.05$) of antioxidant compounds was found in the pomelo residues compared to the pomelo flesh. One of the major antioxidant compounds namely naringin in pomelo peel was stated as a health-promoting compound with the ability to lower the risk of heart disease. However, high levels of moisture from fresh pomelo residues cause it to deteriorate over time, because of the microorganism reactions. Thus, the drying process was recommended as a method of preservation. Conventional hot air oven drying (CD) is known using hot air as a heat source for drying a product while the mechanism of vacuum oven drying (VD) utilized the removal of water molecules by evaporation due to the reduced pressure. However, optimum drying methods or conditions have yet to be investigated. Furthermore, the determination of drying kinetics of conventional drying (CD) and vacuum drying (VD) which, explain the drying behavior of pomelo residues at different drying temperatures has not yet been established. Information on the degradation of the kinetic constant of naringin and its antioxidant capacities in pomelo residue during storage is also limited. Therefore, the overall aims of this research were to investigate the impact of different drying methods (CD and VD) on

the quality of pomelo residues. The selection of optimum drying parameters was based on the capability to retain high total phenolic content (TPC) and contribute to antioxidant capacity (DPPH scavenging activity), where high desirability index (D ≥ 0.80) were obtained from statistical analysis. Furthermore, kinetic modeling was analyzed for both drying methods and degradation of naringin in dried pomelo residues during storage were also identified. Selection of best drying models was referred to the highest value of determination of coefficient (R2) and the lowest value of standard error of estimates (SEE) between drying models. The effects of storage time (12th week at 8̂°C) for both drying methods applied were also compared and the fitness of kinetic degradation of naringin and antioxidant capacities to zero, first and second order was evaluated. The results showed that both drying methods (CD and VD) at different drying temperature significantly affected ($p < 0.05$) the quality of the pomelo residues. Pomelo albedo was found to contain the highest total phenolic content when compared to other pomelo residues. The current findings also showed that VD at 90 °C (VD90) was indicated as the best drying process for pomelo albedo (D ≥ 0.80). The diffusion approach model showed the best fit for CD whereas the two-term exponential model showed the best fit for VD method of pomelo albedo at 50 - 90 °C of drying temperatures. The effective moisture diffusivity (Deff) was shown to decrease linearly over the stated temperature range - from 5.00 x 10-7 to 6.98 x 10-7 m2/s for CD whereas Deff for VD was shown to increase -within the range of 9.86 x 10-7 - 1.30 x 10-6 m2/s. The absence of oxygen was shown to be correlated with the rate of moisture that evaporated from samples to the environment under vacuum condition in VD drying process ($p < 0.05$). During storage, vacuum dried crude extract (VC) attributed to a higher concentration of naringin when compared to freeze-dried crude extract (FC) at the initial week (0th week)($p < 0.05$), however, as storage time increases, the rate of naringin degradation kinetic were found to be lower in FC ($p < 0.05$), indicating higher stability of FC than VC. Naringin degradation can be described in zero, first and second order with the highest value ($R2 > 0.89$) and lowest value of SEE (value less than 0.05). The present study provides insightful information for the post-harvest research, food, nutraceutical, and pharmaceutical industry as a guideline

to produce a new alternative source of higher bioactive compounds from pomelo's albedo.

Keywords : Pomelo, Bioactive compounds, antioxidants, Kinetic models, Drying

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CHAPTER 1
INTRODUCTION

1.1 Overview

Pomelo, pummelo or "Limau Bali" (*Citrus grandis (L.)* Osbeck) is believed to be an ancestor of the Rutaceae family. It is round in shape, greenish in colour, 15-25 cm in diameter, and usually weighs about 1-2 kg (Salihah, 2015). The pomelo is famous across South East Asia because of its nutritional contents and health benefits. In 2014, the Department of Agriculture of Malaysia has reported that 1,066 hectares of pomelo's plantation area lead to the production of 15,281.2 metric tons per year (DOA, 2018). In Malaysia, the demand for the fruit starts is only during Chinese New Year because of the symbolization of bringing continuous prosperity when consumed it. Other citrus fruits that are eaten during Chinese New Year period includes tangerines and oranges. The pomelo fruit is also used regularly for the ritual purposes or consumed freshly while the peel is discarded.

In recent years, many studies have been conducted to determine the physicochemical properties of pomelo fruit and its juice. Some studies have compared the differences in physicochemical properties between the Malaysian varieties of Pomelo fruit Ledang (PO55) and Tambun (PO52) (Buang, 2016). Other studies have reviewed on the high yield extraction method of pomelo juice (Shamsudin et al., 2015), and the effect of storage on the qualities such as textural and physicochemical properties of fresh cut and the whole fruit of pomelo (Niponsak et al., 2011; Sirisomboon and Lapchareonsuk, 2012).

Furthermore, the high antioxidant capacities of pomelo fruit and its by-products have attracted high interest among researchers. The presence of the phenolic compounds (naringin, neohesperedin, hesperidin, coumarins) in pomelo juice (Shahidi and Ambigaipalan, 2015) as an active phytochemical, are believed to be able to prevent or reduce the rate of oxidation and prolong the shelf life (Cohen et al., 2000). In fact, several studies have proven that pomelo byproducts contained bioactive

compounds and showed antioxidant effects (Chang and Azrina, 2017; Jiang et al., 2014; Manorenjitha et al., 2016; Pichaiyongvongdee and Rattanapun, 2015; Pichaiyongvongdee et al., 2014; Toh et al., 2013; Zarina and Tan, 2013). Sultana et al. (2012) added that phenolic antioxidant effectiveness has gained interest for its ability to counteract free radicals, which linked to a variety of diseases.

However, it is well known that fresh fruits residues deteriorate easily and cause significant nutrient degradation due to the cellular respiration and oxidation within a short time. Therefore, they are preferably stored in a dry or low-temperature condition to retain the quality. Quality comprises physicochemical, nutritional and microbial changes during processing and storage. Processing food need to be applied to improve the quality of foods by verifying the safety, retaining the nutritional value and developing a high-quality end product. Thus, drying is a commonly applied method of preservation, which involved removal of water from the foodstuff. However, pharmacological alteration and the quality of fruits can be affected, in terms of chemical, nutrition, and physiological during processing. It is well accepted that the condition of storage and processing give notable effect on the bioactive compound particularly carotenoids, phenolic antioxidants and vitamin C of fruits and vegetables (Gebezynski & Kmiecik, 2007; Sultana et al., 2012).

In this study, drying process includes convective drying (CD), freeze drying (FD), and vacuum drying (VD) were selected based on specific reasons. Conventional hot air oven drying (CD) is known using hot air as a heat source (Perazzini et al., 2015) for drying a product. CD is a very low cost and user-friendly method compared to freeze drying (FD), which requires higher cost and lengthy procedure (Vashisth et al., 2011). However, there are major effects in terms of quality (nutrient compound) and color of the final product (Wojdylo et al., 2014) after drying compared to freeze dried products. Meanwhile, vacuum oven drying is exploited due to the commonly used for preserving heat-sensitive compounds. The removal of water molecules of the vapor phase is expanded and evaporated due to the reduced pressure during vacuum drying (Karam et al., 2016). As a result, it allows limited changes on physicochemical properties and preserving ascorbic acid compound (Oikonomopoulou and Krokida, 2013).

During processing and storage, changes of the particular food can be predicted by a system known as kinetic modeling. Scientists have shown significant interest in this fields. Not only the changes have become the main focus, the mechanism occurred in the process and the statistical evaluation will also need to be measured. Kinetic modeling of drying involved mathematical equations describing the removal of water to predict the dehydration time of the targeted product. Relevant parameters in the mathematical equation are validated by experimental data conducted (Boekel, 1996).

Furthermore, various processing and storage conditions such as exposure to high temperature, light, and oxygen during storage will result in the potential of oxidation (Bouzari et al., 2015). Thus, the condition of the storage is very crucial as we target to retain and minimize the degradation of the target compound.

Previous researchers have reviewed citrus peel as an excellent raw material for the bioconversion into value added product and valuable sources of phytochemical and pharmacological potential (Mahato et al., 2018; Mamma & Christakopoulos, 2008; Negro et al., 2016; Zainol Abidin et al., 2019). Naringin (4'5,7 –trihydroxyflavonone-7-rhamnoglucoside) is one of the most interesting phytochemical compound that naturally occurs in citrus. Naringin exhibits a remarkable spectrum of biological properties including anticancer, cholesterol-lowering, antiapoptotic, antiatherogenic, and metal binding effects and antioxidant activities (Bacanlı et al., 2018; Chen et al., 2016). Recovery of naringin has been discovered in grapefruits (Castro-Vazquez et al., 2016; Jiang et al., 2014; Mäkynen et al., 2013; Xi et al., 2014). The presence of valuable compound possess chemotherapeutic impact from pomelo residues can be fully processed and utilized into value-added product.

Therefore, as drying circumstances and post-harvest regimes may affect the composition of fruit byproducts, the present study aims to identify the effects of different drying practices; freeze drying, conventional drying and vacuum drying on the physicochemical and antioxidant activity of the pomelo byproducts.

1.2 Problem statement

Utilization of waste into wealth have been applied recently, with the exploration on the functionality and identification of health promoting compound in the residues, have been carried out by previous researchers. Despite of wide range of domestic and industrial exploitation, pomelo peel is usually discarded due to the lack of awareness as a sustainable resource of its health-promoting composition from the wastes produced. In Malaysia, the Department of Agriculture has reported that 1,066 hectares of pomelo's plantation area lead to the production of 15,281.2 metric tons per year (DOA, 2018). Based on this number, production of pomelo has increased significantly from 1960 to 2018. Since ~50% of pomelo weight is derived from the peel, approximately 4,213 metric tonnes (RM 25.7 million / USD 6.0 million) of pomelo peel being discarded per year in 2018 (FAO, 2018) without recognizing its possible nutritional value (Ani and Abel, 2018; Czech et al., 2020; Shamsudin et al., 2015) and bioactive compounds. Studies have shown that pomelo peel contains a high amount of bioactive compounds and revealed antioxidant effects (Chang and Azrina, 2017; Jiang et al., 2014; Pichaiyongvongdee et al., 2014; Toh et al., 2013). Previous studies have found the potential of pomelo peel as a source of dietary fiber Mat Zain et al. (2014) and bioactive compounds (flavonoid, phenolic compound, naringin and limonin) (Arumugam et al., 2018; Chang and Azrina, 2017; Pichaiyongvongdee and Rattanapun, 2015; Zainol Abidin et al., 2019; Zarina and Tan, 2013), pectin (Methacanon et al., 2014; Sotanaphun et al., 2012), and essential oils (Chaiyana et al., 2014) with great benefits for health.

However, postharvest pomelo residue containing high moisture content (MC) and easily due to perishable properties. High MC tends to deteriorate pomelo peel as the free water is available for microbes and eventually degrade the nutrient values. Hence, drying is the most common preservation method to prevent the microorganism activity (Geankoplis, 2003; Ghanem et al., 2012). The most popular drying methods are conventional hot air oven drying (CD) and freeze drying (FD). CD is a very low cost and user- friendly method compared to FD, which requires higher cost and lengthy

procedure (Vashisth, et al., 2011). However, there are major effects in terms of quality (nutrient compound) and color of the final product (Wojdylo, et al., 2014). Alternatively, vacuum oven drying (VD) were used for drying citrus peel prior to extraction of phytochemicals and antioxidant (Papoutsis et al., 2017). FD and VD capable to preserve the heat- or oxygen sensitive bioactive compounds. Quality parameters such as texture, shape, chemical changes of the product could be significantly affected by the drying process. The quality of dried end product and its natural phytochemical efficiency (Gümüşay et al., 2015) should be retained after drying. Previous study reviewed the impacts of thermal processing on bioactive compound of citrus peels (M'hiri et al., 2016). However, to the knowledge of author, no studies have done on choosing ideal drying condition for retaining maximum antioxidant capacities of pomelo residues. Therefore, optimization of best drying method to retain the best quality of the final product is highlighted in current study.

Furthermore, drying conditions, dryers and the properties of materials significantly affect the drying kinetics. By using mathematical models, identified parameters can be used for development, optimization and improvement of the product quality (Onwude et al., 2016). Several thin-layer mathematical models are available in literature, but their implementation depends on the type of material to be dried (Ling et al., 2015; Ramírez et al., 2015; Torki-Harchegani et al., 2016). Previous researchers have studied the drying kinetics of different types of citrus using hot air drying method (Torki-Harchegani et al., 2016), pulsed vacuum drying (Wang et al., 2018), infrared-vacuum drying (Salehi and Kashaninejad, 2018), microwave drying (Kesbi et al., 2016) on lemon slices, infrared drying (Xu et al., 2017) on tangerine peel, and only hot air drying (Cantu-Lozano et al., 2013) for grapefruit seeds. Although several studies on drying modelling of citrus have been taken part in literature, there is still limited information exists with various drying techniques (CD and VD) on pomelo residues take places.

Storage analysis is significant to carry out to determine the expiration date and shelf life of a commodity. Pre-treatment such as drying methods could be compared to improve and prevent the spoilage to be occurred and mathematical models of

storage time could be used to predict shelf-life commodities. Previous study supported that storage and processing have shown the significant effect on the related health promoting compounds such as caretenoid, vitamin C and phenolic compounds in fruits and vegetables (Gebezynski & Kmiecik, 2007; Sultana et al., 2012). For example, most previous studies detailed the effects of storage time on quality (ascorbic acid, phytochemicals; e.g. phenolic and flavonoid composition; neohesperidin, naringin, naringenin) for blood orange juice (Remini et al., 2015; Touati et al., 2016; Wibowo et al., 2015) and grapefruit juice (Agudelo et al., 2017; Igual et al., 2010; Moraga et al., 2012; Shahidi and Ambigaipalan, 2015). The mathematical models based on the order reaction (corresponds to zero-, half-, first- and second-order reaction modelling) is used to describe the shelf life mechanism (Ling et al., 2015; Van Boekel, 2008). Result of the kinetic parameters is essential to predict the antioxidant quality change during storage (Kim et al., 2018). Degradation kinetic studies on different drying temperatures during storage have been reported for citrus juices (Igual et al., 2011; Remini et al., 2015; Touati et al., 2016). Nevertheless, degradation kinetic studies were mostly carried out during drying process, but limited during storage period, thus, to the best of our knowledge, no kinetic data were published regarding the degradation kinetics of naringin compound and its antioxidant capacity from pomelo residues (crude extract) during storage.

1.3 Objective

This study is focused to determine the effects of different drying methods on the quality of pomelo residues and to select the optimum drying condition of pomelo residue. The best process is required to have the same quality comparable with the characteristics of freeze-dried pomelo residue. Subsequently, drying kinetics and modeling equations on the pomelo albedo were studied and analyzed. Further on, the kinetic degradation of naringin and its antioxidant capacity during storage condition of pomelo albedo were also investigated.

The specific objectives targeted to achieve are summarized as below

1. To optimize the drying conditions on physico-chemical and antioxidant properties of pomelo residue

2. To analyze the experimental data into the suitable drying model and the effective moisture diffusivity for albedo

3. To fit the mathematical model on the degradation of naringin compound and antioxidant capacity during storage condition for albedo

1.4 Scope of Thesis

This research focused on the potential discovery of choosing the optimum condition for developing a useful byproduct from pomelo residues. The pomelo residues were collected to observe the impacts of different drying temperature (50, 60, 70, 80 and 90 °C) and drying methods (freeze-drying, FD; conventional hot air drying, CD; vacuum drying, VD) onto physiochemical properties and antioxidant properties (total phenolic content and its antioxidant capacities) of pomelo residues namely, flavedo, albedo, lamella and pulp waste. The physicochemical properties include the moisture, color, ash, protein, fat and total phenolic content, and DPPH radical scavenging activity and ferric reducing ability affected by freeze-drying (FD), conventional oven drying (CD) and vacuum oven drying (VD) were discovered. Optimization of drying condition was only discovered for selected pomelo residues(albedo) due to the highest phenolic content discovered compared to other pomelo residues. The pomelo albedo was used to discover the suitable drying model to mimic the drying mechanism of conventional drying and vacuum drying of pomelo albedo. Based on the higher coefficient of determination (R^2) and lower standard error estimate (SEE), selection of the best drying model can be selected. The measurable total phenolic content, naringin and its antioxidant capacities were evaluated during 12 weeks of storage. The kinetic degradation of vacuum-dried crude extract (VC) and freeze-dried crude extract (FC) were compared and evaluated based on zero, first and second order.

1.5 Thesis Organization

The introduction in chapter 1 generally reviews utilization of pomelo wastes which has been discarded without considering the potential benefits, drying processing involve as preservation methods of fresh pomelo residues. Problem statement, the objective of research and its significance that support the contribution of the research are also presented in this chapter. Chapter 2 describes previous studies have been done in pomelo fruits residues including physicochemical properties, and nutritional composition. This chapter also discusses previous works on drying process which affect the quality of pomelo fruits and its relationships. Different drying operation, its significantly effects towards quality and bioactivity of pomelo residues are elaborately discussed. Technical operation, its parameter involved in drying models at different drying operation were also discussed in this chapter. Major phenolic compound presenting most citrus fruits were reviewed and compared. Kinetic modeling involved in different drying methods also were compared. Chapter 3 reviews detailed experimental design and methods in finishing this research. The experimental drying was comprehensively presented in this chapter. In addition, the chapter presents the methods in obtaining all the responses, yield of different parts of pomelo residues of preliminary study, drying treatments, physicochemical composition including color, proximate composition, and antioxidant activity. The method to fit the experimental data of drying kinetics into drying models were carried out. Analysis on major phenolic compound of selected pomelo residues and undergoing storage condition at 8 °C were reported and described. Kinetic models follow the Arrhenius law during storage were reported. Statistical analysis using one-way anova was used to compared either the variation of the results obtained were significant at $p < 0.05$. Chapter 4 presents the findings of research investigations on the objectives mentioned previously. Preliminary study of total yield of different parts of Tambun Pomelo White (PO52) were done to identify which have the highest yield of pomelo residues. Physicochemical properties of pomelo residues were compared based on compound. Selection of drying condition and pomelo residues were discovered and reported. The selected part was undergo different drying methods and identify the effective diffusivity and energy activation

were compared. Kinetic degradation of drying of pomelo peels during storage were compared. Chapter 5 summarize the significant results obtained in the current studies with a several recommendations were given to improve the current study in scope of field.

1.6 Contributions of Thesis

The contributions of this study are many since the pomelo fruit is non-seasonal and non-climacteric fruit which could be planted and harvested all year long. Pomelo residues has been discarded without knowing the presence of potential compound including phenolic compound that are good for human health (anti- cancer, cardiovascular disease, anti-inflammatory and many more). Phenolic composition is known as second metabolites has been reviewed exhibit positive impact to oxidation and capable to prevent chronic disease.

This study if proven successful could provide the alternative drying pomelo residues with retaining the quality particularly on antioxidant activity of the pomelo residues (control: freeze drying). Vacuum drying being the costly dryer and retaining the highest antioxidant capacity compared with conventional drying can benefit the SME and DOA, Malaysia thus realizing the aims of National Agro-Food Policy (2011-2020). Drying kinetics of pomelo residues provide the effects on moisture variation during drying which could contribute for agriculture sector in processing of pomelo residues. Foremost compound of phenolic constituent known as naringin were identified present in pomelo residues. The natural antioxidant activity of the dried crude extract would be retained, and these benefits are considered important to consumers. Retention of antioxidant capacities crude extract of pomelo can be a crucial advantaged where, naringin would be retain higher than FD, thus, enhancing the period of storage and ultimately prolonging the shelf life of pomelo crude extract. In addition, research for these residues should be recommended to follow the directions of ‘waste to gold’ application, by carried out further research explained in following section like physicochemical (color, nutritional composition, antioxidant properties) composition. Not to forget the way to preserve the product, so that, at the

end of the product should maintained the quality that comparable with the control. For instance, the effects of drying processing (varies in drying temperature, drying methods) towards them were discussed thoroughly in following section and become a higher quality product become potential sources for functional foods and other application as well. Thus, the current study could provide an insightful reference for related agencies such as agriculture sectors, waste management and food industries for further actions).

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

Section 2.2 describes the overview of citrus and pomelo production worldwide, different pomelo varieties from different countries and the area cultivation of pomelo fruits and trees. Section 2.3 relates to the physicochemical, nutritional composition and potential benefits of pomelo fruits and its residues. Sections 2.4 reviews on drying methods involved and the effects of drying on the quality of pomelo fruits (section 2.5) and summarizes the effects of drying on physicochemical properties, nutritional composition and antioxidant properties of selected citrus fruits based on the past studies. Lastly, section 2.6 discusses on the mathematical modeling of fruit and summarizes the related models. Meanwhile, summary on effects of storage time and kinetic modeling related were reviewed in section 2.7.

2.2 Overview

Nowadays, consumer demand healthy food and drinks have extended across genders and generations. This growing trend is driven by an increment of consumer desires for healthier lifestyles. For that reason, fruits have commonly become favorable food as they are highly nutritious and delicious. High demand of the citrus fruits such as mandarin (*C. reticulate* Blanco), sour orange (*C. aurantium* L.), lemon (*C. limon*, L. Burn. f.), lime (*C. aurantifolia* Christm), sweet orange (*C. sinensis* Osbeck), grapefruit (*C. paradise* Osbeck), pomelo (*C. grandis* Osbeck) are due to their taste, nutritious and health benefits to human health (He et al., 2012). The composition of these fruits possessed tremendous amounts of bioactive compounds and antioxidant potential, which are correlated to demonstrate the anti-inflammatory, anti-cancer, anti-tumor, and blood clot inhibition actions (Garg et al., 2001; Bacanlı et al., 2018).

The responsible health-promoting compounds in citrus fruits are known as ascorbic acid, phenolics, carotenoids (lycopene and b-carotene), related nutrients (thiamine, riboflavin, nicotinic acid/niacin, pantothenic acid) and folic acid (Ladaniya, 2008). The most abundant crop of citrus fruit trees in the world, with an annual production of approximately 194.4 million tons are oranges (84.7 million tons), mandarins (53.6 million tons), lemons and limes (21.9 million tons) and grapefruit (14.4 million tons) (FAO, 2018). Citrus fruits producing countries (million tonnes) can be seen in Figure 1

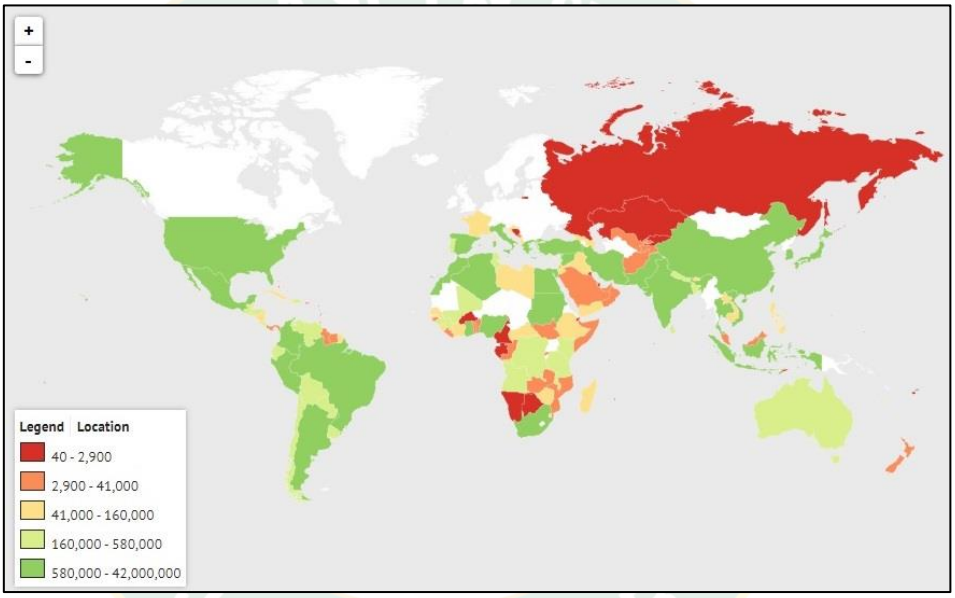

Figure 1 Citrus fruits producing countries (million tonnes);
Source: FAO (2018)

2.2.1 Production of pomelo

The largest kind of citrus fruits is known as pomelo (*Citrus grandis* (L.) osbeck). Pomelo belongs to Rutaceae family, which is known as a native citrus fruit. It is originated from the tropical and subtropical Southeast Asia and Indo-China regions. The world leading production based on planted area of pomelo fruits are summarized in Table 1. It shows that the three largest grapefruit (including pomelo) producing countries are China (10 million tons/year), Vietnam (around 657 660 tons/year) and USA (about 558 830 tons/year). Consistently with the largest cultivated area (179, 823

ha), China is the largest pomelo producer in the world. This indicates that 55.73 tonnes per ha are produced in each year.

Table 1 Top ten pomelo production regions in the world based on volumes produced and area planted**.**

World rank	Country	Production (tonnes)	Area planted (ha)
1	China	10,022,099	179,823
2	Vietnam	657,660	86,370
3	United States of America	558,830	20,113
4	Mexico	459,610	18,823
5	South Africa	445,385	14,472
6	India	257,750	10,572
7	Turkey	250,000	5,182
8	Sudan	234,388	19,382
9	Thailand	219,838	24,664
10	Israel	148,896	2,259

Source: FAO (2018)

Pomelo also has been cultivated in other countries such as Thailand, China, and Malaysia. The varieties of pomelo that can be found in China, Thailand and Malaysia are summarized in Table 2.

Table 2 Pomelo variety of selected countries

Country	Genotypes	Reference
China	• *C.grandis* cv. Wentan • *C.grandis* cv. Liangpingyou • *C.grandis* cv. Huayingshanyou • *C.grandis* cv. Hongxinyou • *C.grandis* cv. Meiweishatianyou • *C.grandis* cv. Gaopuyou • *C.grandis* cv. Shatianyou • *C.grandis* cv. Wanbaiyou • *C.grandis* cv. Dayongjuhuaxin • *C.grandis* cv. 24-14 • *C.grandis* cv. 14-13 • *C.grandis* cv. Chandler • *C.grandis* cv. Dongfengzao • *C.grandis* cv. Zaoshuyou • *C.grandis* cv. Zuoshiyou • *C.grandis* cv. Qiyou • *C.grandis* cv. Guanximiyou • *C.grandis* cv. Menglunzao • *C.grandis* cv. Tongxianyou • *C.grandis* cv. Liboyou • *C.grandis* cv.Linnanshatiaoyou • *C.grandis* cv. Sijipao • *C.grandis* cv. Jintanglvyou • *C.grandis* cv. Shishengyou • *C.grandis* cv. Guanxiangyou • *C.grandis* cv. 28-19 • *C.grandis* cv. Anjiangxiangyou • *C.grandis* cv. Guokuiyou	Xi et al. (2014)

Thailand	Nakhon Pathom Province; • Thong Dee (TD), • Kao Paen (KP) • Kao Nampheung (KNP) Samut Songkhram Province; • Kao Yai (KY) Phichit Province; • Tha Knoi (TK) Chi Nat Province; • Kao Tanggkya (KTG) Nakhon Si Thammarat Province • Pattavee (PV)	Pichaiyongvongdee and Haruenkit (2009); Pichaiyongvongdee et al. (2014)
Malaysia	• Tambun (PO52), • Sha Thing (PO51), • Ledang (PO55) • KK2 (Melo Mas)	Anim (2012)

In Malaysia, currently, pomelo is widely grown in the states of Sabah, Perak, Johor, Melaka, Negeri Sembilan, Pahang, Kedah and Kelantan (DOA, 2018). In Peninsular Malaysia, Perak is the biggest producer of pomelo where the cultivated area is in Kinta District (1,986.4 mt) followed by Kerian District (593.8 mt) and Kuala Kangsar District (389.0 mt). Table 3 shows the production and cultivated area of pomelo fruit in Malaysia. Pomelo grows in humid and hot climate and the peak seasons for harvest are from January to February and August to September in Malaysia (DOA, 2018).

Table 3 Production of pomelo in the state and peninsular of Malaysia

State	Production (mt)	Planted area (ha)
Perak	3,000.74	149.11
Johor	2,717.46	150.50
Melaka	965.01	40.10
Negeri Sembilan	131.36	7.50
Pahang	102.33	12.32
Kedah	67.28	2.70
Pulau Pinang	28.12	3.20
Perlis	15.00	1.00
Kelantan	8.62	1.18
Selangor	3.00	0.80
Total		
Peninsular Malaysia	**7,038.93**	**367.96**
Sabah	7,109.30	440.90
Sarawak	1,131.70	257.30
W.P. Labuan	1.25	0.30
Total	**15,281.18**	**1,066.46**

Source: DOA (2018)

There are few popular varieties of pomelo fruits known in Malaysia, which are Tambun (PO52), Sha Thing (PO51), Ledang (PO55) and Melo Mas (PO56) (Anim, 2012; Shah, 2015). The pomelo fruits also can be called as pummelo, pomelo, shaddock, and Chinese grapefruit, whereas in Malaysia, pomelo also known as limau bali,limau abong, limau besar, limau bol, limau jambua or Bali lemon (Toh et al., 2013). In 2018, Department of Agriculture of Malaysia has reported that 1,066 hectares of pomelo's plantation area lead to the production of 15,281.2 metric tons per year (DOA, 2018). Based on this number, production of pomelo in Malaysia has shown to be increased significantly from 1960 to 2018. It shows that the production and demand of pomelo fruits have increased every year.

High demand for pomelo fruits as it is being processed to make jellies or marmalade in Thailand and used for ritual purposes as it is believed to bring prosperity. In addition, the peel can be eaten as candy or used as a jam after boiling it in syrup. Due to its highly nutritious compounds, people are demanding the pomelo fruits especially during Chinese New Year celebration as the fruits are believed to bring good fortune to the consumer.

2.2.2 Morphology of pomelo fruits

The morphology of citrus needs to be established for grading process, which is done based on the size, volume, shape, weight, and area of projection. Area of projection is the two-dimensional area measurement of a three-dimensional object by projecting its shape on to an arbitrary plane. Furthermore, this knowledge is correlated with the scheme of handling, grading, sorting or packaging process (Safwat, 1971; Peleg and Ramraz, 1975; Salihah et al., 2015). In addition, the most preferred way of the consumer in choosing the fruits is by observing the physical properties of the fruits with similar shape and identical weight. Salihah et al. (2015) have found the interrelation between the mass modeling and pomelo properties, which can be used as a primary measurement of the grading system in the future. In general, the evergreen citrus and cultivars species grow and produce fruit under a wide range of climates and soil conditions have shown a varying degree of success. The pomelo tree is a large shaggy tree includes the thorny irregular branches that can grow until 5 to 15 m in height (Anim, 2012). The trunk is thick (10 to 30 cm) with irregular shape and spreading with a low branches, while the young branches are densely soft and angular with spines (Buang, 2016). As for the leaves, they are 2 to 12 cm wide and have small dots spotted on the leaves, which promotes the dark green color and shiny appearance. Small dots represent the oil glands containing essential oils (Anim, 2012). Pomelo is the biggest citrus fruit and believed to be a precursor of grapefruits. The diameter is 15-25 cm and the common weight is around 1 to 2 kg per fruit. In brief, the pomelo flesh is covered by three morphological regions which are known as flavedo, albedo, and lamella as shown in Figure 2.

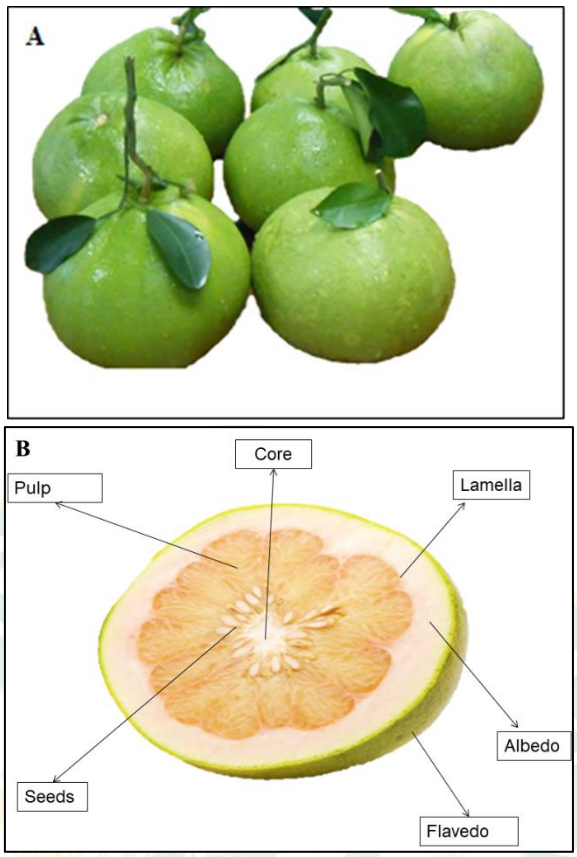

Figure 2 The whole (A) and cross section (B) of pomelo fruits.
Source: Sirisomboon and Theamprateep (2012)

The outermost part is known as flavedo, the exocarp is the outer layer with the green distinct color of epidermis covered by waxy cuticle layer and packed with parenchyma cells beneath it (El-Otmani et al., 2011; Thielen et al., 2015). The second layer of the peel consists of an endocarp, the white spongy albedo and it is thicker than flavedo and lamella. The third layer is the tough thin layer with slightly pinkish color that covers the numerous juice sacs called lamella, or segment membrane (Chang & Azrina, 2017). Eight to nine segments membrane are observed in pomelo fruit (Sirisomboon & Lapchareonsuk, 2012). Generally, the various sizes of seeds are found in the middle of segments which consist of fruit flesh. At the center of the fruit, there is a core that is hollow when the fruit is ripe and holds the flesh covered with lamella. About five and half month after the full blossom, the fruit is ripen and the color is changed from dark green to light green.

During development of the crop, postharvest handling, and storage, the rind is important as it protects the fruits from impairment, insects, and critical temperature. The thickness of the rind depends on the cultivar, the climatic conditions, and the stage of development (El-Otmani et al., 2011). The flesh of pomelo fruit includes juicy pulp, which is commonly eaten fresh or pressed to obtain the juice. In brief, the residue left after extraction of the juice, known as citrus pulp or pomace, represents 49-69% of the fruit by weight. This waste includes segment membranes, peels, seeds, and pulp waste (Li et al., 2006). The citrus pulp contains a high content of water and soluble sugars that can lead to environmental pollution.

Mostly, pomelo has significant peel and segment membrane in comparison with other citrus fruits, which eventually leads to generating the high quantity of pomelo waste. Table 4 reflects the yields of by-product from selected citrus fruits.

Table 4 The yield of by-product from other citrus /selected fruits.

Sample	By-products	Edible part	References
Guangxi pomelo	63.1% (peel)	36.9%	Bai et al. (2013)
Mandarin	16% (peel)	84%	Ayala-Zavala et al. (2011)
Oranges	66% (peel)	44%	Li et al. (2006)

2.3 Physicochemical properties of pomelo fruits

The identification of the morphological and biochemical features of citrus fruit are important in the field of postharvest management based on research and development. Numerous studies have attempted to establish the significance of fruits by-product and gather the information by reviewing their differences and roles in certain applications such as water and diseases treatment, rich of nutritional properties, and the bioactive compound retainment (Bhatnagar et al., 2015; Ledesma-Escobar and Luque de Castro, 2014; M'hiri et al., 2014). The chemical composition of Ledang and Tambun of pomelo juices can be referred in Table 5.

Table 5 Chemical composition of Ledang and Tambun variety pomelo fruit juice

Analysis	Ledang	Tambun
Juice yield (%)	42.2 ± 0.008	38.2 ± 0.004
Total acidity (g/L)*	11.3 ± 0.2	13.43 ± 0.15
pH	3.99 ± 0.02	3.88 ± 0.015
Brix	12.23 ± 0.057	14.17 ± 0.057
Total phenolics (mg/100 mL)	28.67 ± 0.58	46.67 ± 1.53
Clarity	0.49 ± 0.004	0.36 ± 0.006
Colour	75.51 ± 0.29	76.62 ± 0.096
Ascorbic acid (mg/L)	509.13 ± 3.00	537.47 ± 2.89

Source: Shah et al. (2015)

In recent years, there have been many studies that have investigated the physicochemical properties of pomelo fruit and its juices. Some studies have reported differences in physicochemical properties between the Malaysian varieties of Pomelo fruit Ledang (PO55) and Tambun (PO52) (Buang, 2016). Other studies have reviewed the method of high yield extraction of pomelo juice (Shamsudin et al., 2015), the characterisation of hydrodistillated pomelo peel oil (Chaiyana et al., 2014), the effect of storage on the qualities of fresh cut pomelo (Niponsak et al., 2011), textural properties and physicochemical of pomelo fruit following storage (Sirisomboon & Lapchareonsuk, 2012) and the extraction of pectin from pomelo peels using various techniques (Gamonpilas et al., 2015; Methacanon et al., 2014; Sotanaphun et al., 2012).

Previous research on physicochemical of pomelo fruits and peel has been done previously and can be referred in Table 6.

Table 6 Previous research on physicochemical properties of pomelo fruits and peel

Variety of pomelo	Physicochemical properties	References
Pomelo fruit Variety: Ledang (PO55) and Tambun (PO52)	Differences in physicochemical properties between the Malaysian varieties of pomelo (Ledang and Tambun varieties)	Buang (2016)
Pomelo albedo	Pomelo albedo contains 72.62% carbohydrate, 16.13% moisture, 6.27% protein, 3.41% ash and 1.56% fat. - FTIR spectra for cellulose and nanocellulose confirms absorption bands characteristic -Crystallinity index (CrI) of the isolated nanocellulose was found considerably higher than that of cellulose -Water holding capacity (WHC) of nanocellulose was also higher ($p<0.05$) than cellulose	Mat Zain et al. (2014)
Citrus grandis (L.) Osbeck Albedo of three cultivars—Kao Nampung (KNP), Thongdee (TD) and Kao Yai (KY)	Limonin and naringin contents can be reduced by application of sodium chloride (NaCl), $CaCO_3$ concentration and pH levels pretreatments and different drying methods. - water holding capacity and swelling capacity of the dietary fiber pomelo albedo powder better than the commercial cellulose.	Pichaiyongvongdee and Rattanapun (2015)

2.3.1 Nutritional composition

Nutritional value of food includes moisture, protein, lipid content, and crude fibre (Abirami et al., 2014).

Potential benefits of pomelo and its residues

Nutritional composition of pomelo fruits is tabulated in Table 7.

Table 7 Nutritional composition of pomelo fruit

Sample	Pomelo (Flesh)*
Moisture (%)	88.4
Ash (%)	0.5
Fat (%)	0.2
Protein (%)	0.6
Fibre (%)	0.7
Carbohydrate (%)	9.6

*% based on fresh sample

Source: ASEAN Food Composition Data (2014)

Approximately of 50% yields of peels were discovered from a pomelo fruits which produce significant waste each year. In brief, landfilling is not favorable due to several reasons such as high wastage containing high organic content, transportation cost, lack of disposal site which leads to environmental concern (Cantu-Lozano et al., 2013); Tripodo et al., 2004). Thus, identification and utilization of the agricultural by-products including pomelo fruits are becoming a current trend to intensify and exploit the value added from the waste. According to Wadhwa and Bakshi (2013), the dried citrus pulp could be used as a cereal substitute in livestock feed. Such feed contained a concentrate mixture due to high net energy and suitable for lactating dairy cows.

Furthermore, the use of pomelo waste has been reviewed for production of renewable, low cost and sustainable adsorbents for water treatment applications by Bhatnagar et al. (2015). Pomelo albedo also has potential as a source of fiber (Abirami et al., 2014; Mat Zain et al., 2014; Pichaiyongvongdee and Rattanapun, 2015) which could be considered as a nutritious by-product.

In addition, utilization of 'waste' into 'gold' or healthy beneficial product has been trending in recent years to fully utilize and produce zero waste at the same time

encourage cost and place reduction for waste management. For instance, the peel comprises of an active phytochemical compound (antioxidant compound) known as phenolic, which is capable to prevent or reduce the rate of oxidation and prolong the shelf life of product (Zarina & Tan, 2013; Cohen et al., 2000).

In fact, several studies proved that pomelo by-products contained bioactive compounds and showed antioxidant effects higher than the pulp (Chang & Azrina, 2017; Jiang et al., 2014; Pichaiyongvongdee et al., 2014; Toh et al., 2013; Zarina & Tan, 2013). Thus, the yield of phenolic present in the product can be analyzed by measuring the total phenolic content. Meanwhile, the ability of the bioactive compounds (phenolic constituents) can be assessed by antioxidant capacity analysis in the following section.

2.3.2 Total phenolic content and antioxidant capacity

Total phenolic content is an assay to measure the phenolic antioxidants or phenolic content that present in the dried fruits peel. Folin-Ciocalteau (FC) assay is commonly used to determine total phenolic content (Singleton and Rossi, 1965; Ismail et al., 2013). This particular method is capable to produce predictable results on a wide range of phenolic. In addition, the advantages of FC assay are the simplicity and can be used to characterize, properly control and standardize the variation of botanical samples (Prior et al., 2005; Huang et al., 2005; Shahidi & Ambigaipalan, 2015).

However, several disadvantages have been identified such as the present of certain substances that can interfere the FC reagent and provide elevated apparent phenolic concentration (Prior et al., 2005; Ismail et al., 2013). These substances include inorganic and non-phenolic organic substances from the products. Therefore, it is considered as not specific to phenolic compounds alone.

The principle behind this assay is the promotion of electron transfer pathway by translocation of electrons from the antioxidant compounds (i. e. reducing constituent, phenolic) to the molybdenum (Magalhães et al., 2008; Ismail et al., 2013). It is called as an electron transfer-based assay, which measures the antioxidant capacity of antioxidants in reduction of oxidants. The degree of color changes from yellow to blue can be observed by using spectrophotometer at 750-765 nm when the reduction of molybdenum occurred. This is correlated with the concentration of

antioxidant from the products. Gallic acid can be used as phenolic standard and compared with the extract from the product in order to know the degree of reducing capacity (Ismail et al., 2013). Therefore, the result is represented as Gallic acid equivalent (GAE) per amount of sample.

Captivatingly, numerous studies have shown that citrus contained significant amount of total phenolic content, regardless of the variety of citrus fruits. Table 8 shows the total phenolic content of selected citrus fruits. Various citrus fruits showed different levels of phenolic content. This is consistent with the phenolic compound in different parts of the fruits due to the places of cultivation, processing operation involved and parts of the fruits analyzed (Abdullah et al., 2012; Chang and Azrina, 2017; Ramful et al., 2010; Ramful et al., 2011). These findings have attracted the interest of the researchers due to the benefits of these bioactive compounds to human health.

Table 8 Total phenolic content (mg GAE/g FW) of selected citrus fruits

Sample	Part of fruits		Reference
	Flesh	Peel	
Pomelo from Ledang	NA	132.44	Chang & Azrina (2017)
White Tambun Pomelo	*105.75	NA	Lim and Loh (2016)
Kaffir Lime	*74.22	NA	
Lime	*117.78	NA	
Calimansi	*100.92	NA	
Pomelo Thai	*6.21-12.77	NA	Pichaiyongvongdee et al. (2014)
Ledang and Tambun variety	# 385.3(Ledang) 497.7 (Tambun)	NA	Shah (2015)
Pomelo from			Toh et al. (2013)
Tambun White	4.07	0.71	
Tambun Pink	3.01	0.62	

*mg GAE/g DW

Note: NA: not available; # :GAE mg/L

2.3.3 Phenolic composition

Phenolic composition, commonly known as secondary metabolites is highly important in scavenging free radical and plays various medicinal and beneficial physiological to human. The ability of the phenolic contents to promote an excellent state of health in human has constantly gaining interest (Shahidi & Ambigaipalan, 2015). Phenolic compounds also known as polyphenols; a variety of bioactive compounds that are divided into several classes which are flavonoids, hydroxybenzoic acids, anthocyanins, proanthocyanidins, stilbenes, and lignans (Zou et al., 2016). The most studied phenolic compounds in existing literature among the citrus fruits are flavonoids, phenolic acid, and coumarins (Castro-Vazquez et al., 2016; Jiang et al., 2014; Pichaiyongvongdee and Haruenkit, 2009; Zefang et al., 2016). In order to identify and evaluate the phenolic compounds traditionally, it is time consuming and high labor intensity (Zhang et al., 2015). On the other hand, nowadays, the application of

high performance liquid chromatography (HPLC) enables the detection of a single compound in a complex matrix of food which contributes to the antioxidant activity (Esin Çelik et al., 2014). Consecutively, reduction of cost and time has been observed due to the purification process of each single compound is no longer needed.

The bioactive compounds of citrus plants are varied and diversed based on their origin, species, and different tissues. In existing literature, flavonoids, phenolic acids, and coumaric components were commonly studied in citrus fruits (Zou et al., 2016). For instance, the main phenolic found in pomelo fruits from six different cultivars of Thailand were naringin and naringenin that potentially had antihyperlipidemic activities, which capable of reducing cholesterol level (Mäkynen et al., 2013). Meanwhile, studies have found that pomelo peel contained flavanones compounds known as naringin, neohesperidin, limonin which contributed to the bitter taste of citrus fruits (Chinapongtitiwat et al., 2013). A similar observation was found in the study conducted by Xi et al. (2014), which the predominant composition of phenolic was naringin (2186.47-9871.69 mg/kg fresh weight) in 28 Chinese local pomelos. In addition, the summary of major phenolic compositions presented in the citrus fruit was shown in Table 9.

Naringin, hesperidin, and neohesperidin seem to be the major phenolic compounds in the citrus fruits. Each of phenolic compound gives diversity of response of antioxidant activity. For example, Nakao et al. (2011) reported antioxidant activity of flavonoids (naringin, naringenin, hesperidin, quercetin and rutin) by inhibiting the body's oxidant enzymes, improving the antioxidant compounds activity, directly scavenging reactive oxygen species (ROS), capable of preventing the in vitro lipid oxidation process and reducing the quality formation of peroxide in vivo. In addition, Dai and Mumper (2010) showed that phenolic acid affected the free radical scavenging effect at a different levels based on the dehydrogenation capacity of a hydroxyl group and the effect of ortho substitution on a benzene ring. Meanwhile, the coumarins showed strongly antioxidant activities due to the presence of phenolic hydroxyl groups in chemical structure, which tends to reduce the cellular free radical production (Lin et al., 2008; Tyagi et al., 2005).

Therefore, the phenolic profile is important to be identified as it can be used as references for the exact amount of phenolic in the samples. Furthermore, we can conclude that with diverse of origin, parts of tissues, and types of citrus fruits contain different level of antioxidant content. Thus, it is important to identify and quantify the phenolic composition that presents in particular product.

Different types of antioxidant compounds strongly related to chemical composition and contribute in a different way of antioxidant activities. Correlation between antioxidant compounds and antioxidant activities can be used as a tool to determine whether antioxidant activities associated directly with the antioxidant compounds. However, understanding of the chemical reactions involved in antioxidant analysis is compulsory to accept the rationality behind antioxidant activities. Therefore, the chemical reaction of free radicals effect and how antioxidant reacts when oxidation occurred were explained in the following section.

Table 9 Summary of major phenolic composition present in the citrus fruits

Citrus fruit/Variety/Part of citrus	Identified phenolic compound	Quantity (unit)	Reference
Pomelo from Vietnam • Peels	Naringin	17.10 – 43.30 mg/g sample	Hung et al. (2020)
	Hesperidin	0.13-0.59 mg/g sample	
White and pink grapefruits (Citrus paradise) from Valencia areas (Spain) Related part involved: • Peels	naringin	0.25-18.24 mg/g naringin equivalent	Castro-Vazquez et al. (2016)
	isonaringin	0.22-13.92 mg/g naringin equivalent	
28 Chinese local pomelo (*Citrus grandis* Osbeck) Grapefruit (*Citrus paradisi* Macf.) from Chongqing, China. Related parts involved: • Peel (flavedo/albedo) • Pulp (segment epidermis and juice vesicle)	Naringin	2186.47-9871.69 mg/kg FW(peels) 734.61-4166.19 mg/kg FW (pulps)	Xi et al. (2014)
	Neohesperidin	11.53-7011.15 mg/kg FW (peels) 1.93-3121.72 mg/kg FW (pulps)	
Pomelo of Hua Ju Hong (HJH) variety from Zhejiang province, China Related part involved: • Peels	Narirutin	1.30-7.81 mg/g	Jiang et al. (2014)
	Naringin	11.89-90.58 mg/g	
	Neohesperidin	14.00 -112.77 mg/g	

Table 2.9 (Continued)

Citrus fruit/Variety/Part of citrus	Identified phenolic compound	Quantity (unit)	Reference
Pomelo from Tambun (PO52) and Ledang (PO55) varieties from Malaysia. Related part involved: • Pulp (Juice)	**Hydroxycinnamic acids**		Shah et al. (2013)
	Cholorogenic acid	1.60-2.85 mg/100mL	
	Caffeic acid	0.09-0.11 mg/100mL	
	Coumaric acid	1.33-1.44 mg/100mL	
	Flavanones		
	Naringin	13.09-26.76 mg/100mL	
	Hesperidin	3.57-5.39 mg/100mL	
	Narirutin	3.00-7.19 mg/100mL	
Pomelo (*Citrus grandis* L. osbeck) from Thailand Related part involved: • Pulp (Juice)	Naringin	2.34-41.29 µg/mg dry weight of extract	Mäkynen et al. (2013)
	Naringenin	7.39-29.52 µg/mg dry weight of extract	

Note: The value of the identified phenolic compounds was presented as a range with the minimum and maximum value detected from the selected citrus fruits.

2.3.4 Antioxidant capacity

Antioxidant activity is the ability of antioxidant compound (phenolic constituent) measured by neutralizing free radicals, preventing other oxidative damage, reducing and preventing lipid peroxidation in order to maintain the cell structure and biological function (Bravo, 1998; Zou et al., 2016).

The activity relates to the mechanism of other biological functions such as anti-cancers, anti- inflammation, anti-aging, anti-diabetic, and antihyperlipidemic (Lim & Loh, 2016; Mäkynen et al., 2013; Cai et al., 2004; Ke et al., 2015). Fascinatingly, antioxidant activity has been universally related with the prevention of many chronic diseases, such as cancer, diabetes, cardiovascular disease (Yu et al., 2005; Lim & Loh, 2016; Rajendran et al., 2014; Zou et al., 2016). Consequently, exploitation of natural antioxidant from by-product and biomass from fruits is highly recommended as it is significant to human health.

Recently, antioxidant capacity has been considered as a significant indicator of the beneficial effects from plant foods to human health (Prior & Wu, 2013; Zou et al., 2016). Thus, it is important to evaluate the antioxidant activity from citrus fruits by selecting the relevant methods to analyze it. Numerous studies have been reported on the variety of antioxidant activity evaluation methods for plant samples. Majority used these in vitro methods known as 1,1-diphenyl-2-pierylhydrazy (DPPH) (Abd Ghafar et al., 2010), 2,20-azino-bis(3-ethylbenz thiazoline-6-sulfpnic acid) (ABTS) (He et al., 2012), oxygen radical absorbance capacity (ORAC) (Mäkynen et al., 2013), ferric reducing-antioxidant power (FRAP) (Chang & Azrina, 2017), and trolox equivalent antioxidant capacity (TEAC) (Mäkynen et al., 2013) for citrus fruits. For instance, the antioxidant capacity of different varieties of pomelo fruit is summarized in Table 10 using DPPH and FRAP assay. It is observed that selected assays are capable to measure the antioxidant activities.

Table 10 The antioxidant capacities of different varieties of pomelo fruits**.**

Sample	Parts of fruits	DPPH (%)	FRAP (mM Fe^{2+}/g FW)	References
Pomelo from Vietnam Da Xanh	Peels	13.70 – 43.30	NA	Hung et al. (2020)
Pomelo from Ledang Variety, Malaysia	Flavedo	166.30 (µg/ml)	307.59	Chang and Azrina (2017)
	Albedo	168.00	245.88	
	Lamella	167.45	136.84	
Pomelo from Tambun (White)	Peels	NA	141.25 mM Fe(II)/g dry weight	Lim and Loh (2016)
Pomelo from Taiwan, China	Peels	35.93-55.53	0.87-1.70	Xi et al. (2014)
	Pulp	29.31-58.13	0.90-1.76	
Tambun White	Peel	NA	1.01 mmol Fe(II)/100g FW	Toh et al. (2013)
	Pulp	NA	0.6	
Tambun Pink	Peel	NA	0.65	
	Pulp	NA	0.51	
Pomelo from Bangkok Thailand				Mäkynen et al. (2013)
Kao-Yai (KY)	Pulp	NA	443.56	
Thong-dee (TD)		NA	345.78	
Kao-Tangkwa (KT)		NA	395.22	
Kao-Numpueng (KN)		NA	616.89	
Ta-Koi (TK)		NA	386.33	
Tubtim Siam (TS)		NA	377.44	

Overall, these methods are simple and rapid, however the result is highly influenced by various factors such as different processing and operational conditions involved which include pH, action time, types of biaoctive compounds and their interaction (Zou et al., 2016). Therefore, in this study, the potential of phenolic compounds was measured by measuring the in vitro antioxidant activity such as radical scavenging effect and ferric reducing antioxidant power (FRAP).

2.3.4.1 Radical scavenging effect (DPPH)

DPPH is a stable organic nitrogen radical containing an unpaired electron (Ismail et al., 2013). This DPPH compound (Figure 3) is used as an indicator that can receive hydrogen radical or an electron to become a stable diamagnetic molecule in radical scavenging activity (Liu and Tsai, 2012).

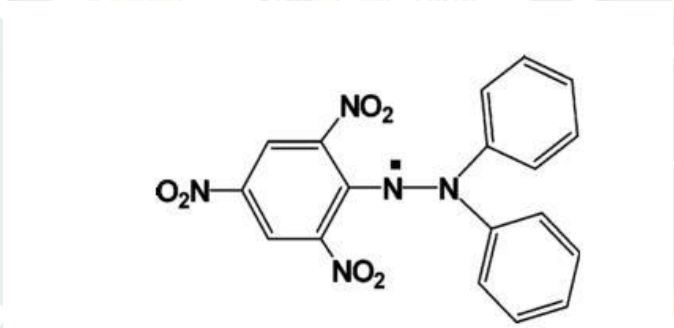

Figure 3 Structure of DPPH**.**

Source: Huang et al. (2005)

Technically, this estimation is simple, rapid and highly sensitive. However, it does not necessarily represent antioxidant compound that corresponds to the real oxidative degradation (Tiveron et al., 2012; Chang & Azrina, 2017). In the present study, the more antioxidant compounds presented in the extract as indicated by the formation of yellow color, the more reduction reactions of DPPH that initially was violet color occurred due to the donation of hydrogen atoms from antioxidant compounds (Abd Ghafar et al., 2010). The yellow color produced can be detected by using UV-Vis spectrometer at wavelength 515 nm (Barbosa-Pereira et al., 2014). Table 2.11 describes the conditions of scavenging effect such as the solvent used to extract the targeted compounds, incubation time, absorbance used, and the result obtained

using DPPH assay from the literature. DPPH is considered as electron transfer based reaction, where it reacts faster than the slow mechanism of hydrogen atom abstraction in strong hydrogen-bond compound (Barbosa-Pereira et al., 2014). This assay involves a simple handling technique and the color changes can be examined using a spectrophotometer (Prior et al., 2005; Ismail et al., 2013). However, the DPPH assay do have some disadvantages such as when big or complex antioxidant molecules involved, the reaction is slower or zero because the fact that radical is more suitable on simple and small scavenging molecules.

Table 11 Condition of scavenging effect of previous study on citrus fruits

Sample	Solvent used/ incubation time	Concentration of DPPH used	Absorbance (nm)	Result of DPPH (%)	References
Lime waste	DMSO/15 min	40 µg/mL	540	175-683 µM Trolox equivalent/g DW	Esparza-Martínez et al. (2016)
Grapefruit peels	Methanol/25 min	0.06 mM	515	25 – 122 mg Trolox per g of dry weight	Castro-Vazquez et al. (2016)
Pomelo peels from China	Methanol /30 min	0.004%	517	40-90	Jiang et al. (2014)
Local pomelo from China	Methanol/25 min	63 µM	517	(35.93-55.53)(Peel) (29.13-58.13)(Pulp)	Xi et al. (2014)
Pomelo pulp from Thailand	Ethanol/	0.2 mM	515	0.41-13.77 mg ascorbic acid/gram dried extract	Mäkynen et al. (2013)
Pomelo peel from China	Methanol	0.2 mM	517	20 - 99	He et al. (2012)

Furthermore, reversible process can occur between DPPH and some compounds (eugenol) and lead to false low antioxidant capacity of samples (Huang et al., 2005; Barbosa-Pereira et al., 2014). In addition, the presence of caretenoid compound can affect the DPPH result as it is able to absorb at the same wavelength (515 nm). However, this assay was used in this study by considering it as a rapid, robust, and simple method. This assay is able to estimate the ability of antioxidant from the food to reduce DPPH radicals. Antioxidant activity can be further expressed as equivalent to the concentration of these known standards (Ismail et al., 2013).

2.3.4.2 Ferric Reducing Antioxidant Power (FRAP)

FRAP originally used to measure the reducing power in plasma, however, it has been adapted in botanicals as one of the antioxidant assays. FRAP assay is commonly used to quantify the overall reducing ability of antioxidant compounds in a redox-linked colorimetric reaction from the plant, which acts as a reducing agent. Briefly, this assay is used to measure the reduction of yellow ferric (III) 2,4,6 – tripyridyl–*s*-triazine complex $[Fe(III)\text{-}(TPTZ)_2]^{3+}$ to ferrous $[Fe(II)\text{-}(TPTZ)_2]^{2+}$ (Benzie & Strain, 1996), a navy blue color complex by antioxidants in acidic medium. The chemical reacion of the FRAP can be referred in Figure 4. The more intense the blue color observed means the more ferric (Fe^{3+}) ion has been reduced to ferrous iron (Fe^{2+}). Therefore, the total reducing capability of antioxidants can be measured using this assay (Guo et al., 2003).

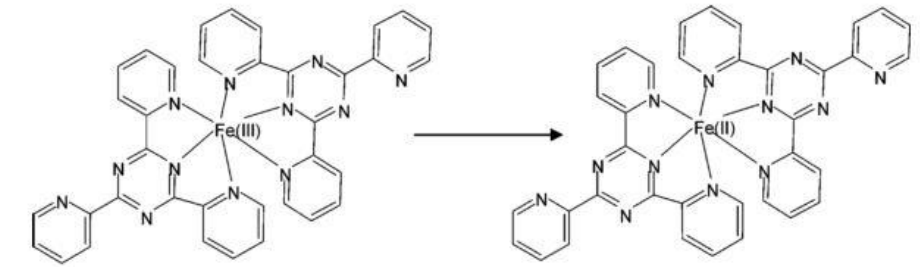

Figure 4 Reducing activity from Ferric^{3+} to Ferrous $^{2+}$ compound**.**

Source: Prior et al. (2005)

It is commonly used because it is easy to be standardized and relatively simple (Prior, et al., 2005). In addition, this assay is inexpensive, the results are highly reproducible and the reagent and instrument used are simpler compared to other assays (Benzie & Strain, 1996; Ismail et al., 2013). In terms of reaction time, typically it only takes around 4 min (Magalhães et al., 2008), however for pholyphenols constituents, longer time (< 30 min) was observed as the compounds react slowly during reaction (Barbosa-Pereira et al., 2014). This situation depends on the time scale of analysis where it can produce different results (Prior et al., 2005). In addition, no reaction is occurred between compounds which acts as hydrogen transfer (such as thiols) in FRAP assay. However, in comparison with other tests of total antioxidant power, the FRAP is preferable to be used due to its simplicity, low cost, and does not require specialize equipment and it was used in this study. In conclusion, based on the information on the antioxidant compound and its capacity of the citrus fruits, it is known that the citrus by-product can be applied in other applications such as feedstock and supplements.

2.4 Drying process

Drying is one of the commonly applied methods of preservation by reducing the water content from the foodstuff. The principle reason of this application is to prevent the microorganisms from multiply, decay or promote undesired changes, which causes food spoilage in the absence of sufficient water (Earl & Earl, 2004; Geankoplis, 2003; Ghanem, et al., 2012). When water is removed from the food, an unbalance pressure occurred within the product, in due course, contracting stress produced and caused shrinkage process (Bejar, et al., 2011). In other words, several drying techniques may cause pharmacological alteration and affect the quality of fruits, in terms of chemical, nutrition, and physiological. Various methods of dehydration commonly applied to preserve fruits and vegetables, which are known as heated air-drying, solar drying, microwave drying, osmotic dehydration, foam-mat, spray-drying, spouted bed drying and freeze drying.

The most popular drying methods are conventional hot air drying and freeze-drying. Conventional hot air (oven) drying techniques are very low cost and easily practiced compared to freeze-drying which is more costly and has lengthy procedure (Vashisth, et al., 2011). However, there is a major effect in terms of quality (nutrient compound) and color of the final product (Wojdylo, et al., 2014) particularly during conventional hot air drying, meanwhile the freeze-drying process resulted in minimal changes on the quality. As for vacuum drying commonly applied for preserving the heat-sensitive compounds because of the removal of water molecules of the vapor phase was expand and evaporates due to the reduced pressure (Karam et al., 2016).

In brief, drying can be done by many means for instance by conventional methods, freeze drying, vacuum oven drying methods. Different drying methods displayed different approaches towards the product. Table 12 shows the main characteristics of different drying methods. Diverse types of drying methods include different medium either in vacuum condition or air atmospheric pressure condition, which relates the operation variables for each drying method. Operation variables mainly consist of drying temperature, velocity, relative humidity for oven drying while the physical properties such as thickness, geometry or density of product need to be properly controlled. Freeze drying has shown similar heat transfer mechanism in terms of internal and external condition

Table 12 Main characteristics of different dehydration methods.

Operation	Drying medium	Operation variables	Main Heat Transfer Mechanism	Internal Mass Transfer Mechanism	Other characteristics
Freeze drying	Vacuum	Medium: Pressure, Temperature Product: Thickness, geometry, density	Internal : Conduction External : Radiation, conduction	Vapor diffusion	Sublimation Subfreezing Temperature
Hot air drying (conventional drying)	Air at atmospheric pressure	Medium: Temperature, Velocity, Relative humidity (RH) Product: Thickness, geometry, density	Internal: Conduction External: convection	Vapor/ Liquid diffusion, capillarity, hydrodynamic flows	-
Vacuum oven drying	Vacuum	Medium: Pressure, temperature, RH Product: Thickness, geometry, density	Internal: Conduction External: radiation and conduction	Liquid/ Vapor diffusion	Possible boiling depending on conditions

Furthermore, different drying methods have their advantages and disadvantages during the drying process, which involved the effect of drying time and the quality as observed in Table 13. Therefore, particular drying process might only be suitable for the particular product to sustain and improve the quality of it.

Table 13 General features of different drying methods involved.

Dryer type	Advantages	Disadvantage
Freeze drying	High nutritional and sensorial quality; no restriction on particle size; utilization of low temperatures.	Slow and expensive process. Vacuum freeze drying is expensive in terms of capital costs and operating costs due to very low vacuum required at very low temperature. Drying times are long.
Tray drying	Simple and continuous process; low operating cost.	Nutritional quality loss; shrinkage; long drying time.
Vacuum oven	Drying chamber is operated at reduced pressure or temperature. Evaporation occurs at low temperature.	Need to maintain high vacuum; expensive. However, absence of drying medium in the vacuum drying chamber disables convective heat transfer but enhances mass transfer at low temperatures.

Sources: Mujumdar (2004); George and Cenkowski (2009)

This information is important to select the ideal drying process to reduce the destruction of the quality during a drying process. Thus, to understand the operation involves behind the drying process, explanation is provided in the following section. In this study, freeze drying, conventional hot air drying and vacuum drying were selected to compare the effects of proposed drying on the quality of pomelo residues.

2.4.1 Conventional hot air drying (CD)

Hot air drying method is a conventional drying method using hot air as a heat source (Perazzini et al., 2015). This kind of drying method is popular based on low-cost process, however, longer drying time is required to have a complete dry. In addition, it also implies oxygen exposure and high temperatures, which may affect physicochemical properties of a product, especially through oxidation and pyrolysis (Nunes et al., 2016). The moisture is transported by liquid or vapor diffusion, surface diffusion, hydrostatic pressure gradient or difference or combination of these within the solid food (Karam et al., 2016). Basically, in air drying, the wet surface material (tray) meets the heated air and the heats are being transferred by conduction. The moisture from product is migrated to the material and transported away by air convection. In brief, air drying consists of 2 stages. At the first stage, free water of the product moves to the surface products and easily evaporates by vaporization. Next, when the drying proceeds, the moisture from the solid materials becomes more viscous and harder to evaporate (it take times for internal moisture to move to the surface) (George, & Cenkowski, 2009) and results in declining of drying rates.

Drying temperature also plays an important role in drying process. Temperature is the most frequently measured variables in process engineering. Ding et al. (2017) explained that temperature has a strong effect on the Maillard reaction mechanism and color changes. Several studies reported on drying condition using conventional hot air drying is shown in Table 14.

Table 14 Condition of conventional hot air drying on citrus fruits based on previous studies.

Sample	Temperature (°C)	Duration (hr)	References
Gallega "lime fruits" (extractables & non extractables)	60, 90, 120	NA	Esparza-Martínez et al., (2016)
Pomelo PO5 (Pink flesh) albedo	50	48 hr	Mat Zain et al. (2014)
Citrus hystrix, Citrus maxima	65	24h	Abirami et al. (2014)
Fiber Powder from Lime Residues	60	NA	Jongaroontaprangsee et al., (2014)
Pomelo from China	50	NA	Ding et al. (2013)

2.4.1.1 Quality of product affected by conventional drying

The quality of product includes the physicochemical, nutritional composition, and antioxidant properties which tend to undergo several modifications during the drying process. The premium quality of end product is highly preferable with maximum retention of nutritional compound and low moisture available for spoilage. In addition, quality of food containing phenolic compound that can exhibit health benefit such as prevention of human diseases (cancer and heart disease) (Shahidi & Ambigaipalan, 2015). However, the quality of food which includes nutritional quality can be affected during drying process (Agudelo et al., 2017; Dhuique-Mayer et al., 2007; Igual et al., 2010). Food quality includes microbial properties, physical properties, enzymatic action, lipid oxidation and chemical reaction of commercial dehydrated products have been reviewed in general (Megías-Pérez et al., 2014; Sablani, 2006). Apart from chemical and physical changes, major loss of nutritional value, vitamin and other compounds are observed (Marey and Shoughy, 2016). This is due to the degree of solubility in water, degradation of certain compounds which are sensitive to heat and oxidation, enzymatic oxidation, and metal on catalysis. Furthermore,

sugar-amine interaction (Maillard reaction) can also cause the loss of nutritional quality during drying and storage (George, & Cenkowski, 2009). Several changes of the food quality involved during thermal degradation are summarized in Table 15.

Table 15 The main effects of thermal drying

Effects	Explanation
Microbial changes	Destruction of cell membranes, leading to denaturation of proteins and death of cells.
Physical changes	Caramelization is promoted by direct heating of carbohydrates (sugar and sugar syrups) and melting; leading to changes in color and flavor.
Enzymatic reactions	Destruction of amino acids and cross linking reactions between amino acids, leading to changes in protein functionality and loss nutritive value.
Lipid Oxidation	When unsaturated lipids are exposed to the excess air present in combustion gases, complex volatile oxidation compounds are formed and cause rancidity, which decreases the quality of foods containing lipids and those in which oils are ingredients.
Chemical reactions	Gelation of starch and hydrolysis.

Sources: Mujumdar (2004) ; Omolola et al. (2017)

The effects of thermal degradation on quality of the product can be used as a reference to determine of a chemical composition is affected by the drying process. Therefore, an ideal drying condition should be identified and controlled effectively to maintain the quality of the end product. Thus, numerous studies have been done on the effects of thermal or drying the quality (physicochemical, nutritional composition and antioxidant properties) (Abirami et al., 2014; Mat Zain et al., 2014; Rafiq et al., 2019) which can be referred to the following section.

2.4.1.2 Effects of drying methods on nutritional composition

Post- harvest plants commodities are composed of water, carbohydrates, proteins, lipids, vitamins, minerals and constituents, which contribute to flavor and aroma. Significant changes will definitely occur during post harvested which affect the quality attributes of the stored products. The changes of nutritional composition affected by different drying methods can be observed in Table 16. The table describes different drying temperatures at a particular time of citrus fruits. Most of the citrus fruits have a major increased in fibre composition compared to other nutritive composition. It could be due to the heat treatment applied and resulted in breakdown of pectic substance and increased in soluble pectin that contributed to the fibre content (Valero and Serrano, 2010).

2.4.2 Freeze drying

Freeze-drying (FD) is a lyophilisation process or known as soft dehydration technique. It incorporates low pressure and low temperature to encourage the sublimation of water from solid to vapor, which represent the ideal process for the preservation of high-value dried products (Karam et al., 2016). This process is well known for its ability to retain product quality (color, form, flavor and nutritional value) similar with its original (fresh) form higher than other drying process, due to the low processing temperature (lower than 0 °C) and the absence of air oxygen during processing, which prevent degradation (Azman et al., 2019; Karam et al., 2016; Karaman et al., 2014; Strummilo & Adamiec, 1996; Vanamala et al., 2005). Other studies also have used freeze dried as a control which represent the fresh produce from oranges

(Chen et al., 2011) comparing with hot air-drying methods at different drying temperature.

During FD, the internal moisture removal occurs in two stages. First stage is sublimation process of ice crystal ensued by the second stage where the remaining unfrozen water undergoes desorption process. The process is operated under vacuum condition with the supplied of controlled heat to stimulate sublimation process (Oikonomopoulou & Krokida, 2013). The details of freeze-drying process is tabulated in Table 17.

Table 16 Effects of conventional drying on proximal composition of citrus fruit.

Sample	Operation	Moisture (%)	Ash (%)	Fat (%)	Protein (%)	Fibre (%)	Carbohydrate (%)	References
Pomelo PO5 (Pink flesh) albedo	Conventional drying, 50°C for 48 h	16.13	3.41	1.56	6.27	Cellulose (21.29%)	72.62	Mat Zain et al. (2014)
Citrus hystrix (peel)	Conventional drying 65°C, 24h	8.66	6.43	3.63	8.04	35.35	46.55	Abirami et al. (2014)
Citrus hystrix (Flesh)		7.2	5.14	5.24	8.23	24.19	57.21	
Citrus maxima (red/peel)		8.12	5.42	3.7	8.56	33.16	49.16	
Citrus maxima (red/pulp)		8.45	4.13	5.45	8.12	21.3	61.02	
Citrus maxima (white/peel)		8.05	5.15	4.12	8.77	32.98	49.98	
Citrus maxima (white/flesh)		7.78	4.24	5.23	8.09	21.19	61.25	

Table 17 Freeze drying process

Steps	Explanation
Preparation	a) To aid drying process, pieces of material should have large surfaces and thin uniform cross-sections, or should have spherical or thin wedge shapes. b) Others are pretreated with sulphur dioxide as an inhibiting agent to prevent the browning reaction.
Pre-freezing. (ice-crystallization)	a) The materials are prepared and frozen at a low temperature. b) All cellular materials contain large quantities of "free water". Dissolved organic and mineral substances in the inter and intra-cellular spaces; "remnant water"- described as "bound" to macromolecules either by electrostatic forces or by hydrogen bonds. c) On freezing, ice crystallizes out of the free water quite easily but generally not out of the bound water. d) The free water is therefore eliminated by evaporation at the beginning of secondary drying. e) By contrast, most of the water bound by electrostatic forces, being difficult to remove, remains in the dry product e.g probably about 1% moisture in freeze dried clls. f) During the process of crystallization, the ice is surrounded by a continuous network of interstitial fluid containing all the dissolved and suspended compounds, which become more concentrated as freezing continues. g) In the end, the whole mass should become rigid, forming a eutectic*****. h) Unless a eutectic is formed, there is a tendency towards denaturation, because the presence of concentrated electrolytes is damaging to cells and proteins. i) Ideally a rigid eutectic phase is necessary also to ensure that primary drying is by sublimation & not combined with partial evaporation from a liquid state.
Primary drying	The ice crystals formed on freezing are sublimed by vigorous and then gentle heating, usually under vacuum.
Secondary drying	With the disappearance of the ice the residual moisture is desorbed at around ambient temperature under high vacuum.
Conditioning and Rehydration	At sufficiently low moisture content the vacuum is broken with a dry inert gas, and the product is then packed and stored.

Notes: *eutectic: homogenous mixture of substances that melts or solidifies at a single temperature that is lower than the melting point of either of the constituents.

2.4.3 Vacuum oven drying

Vacuum oven drying involves removal of water at a lower temperature (~30°C) suitable for dehydration of heat sensitive products (Oikonomopoulou & Krokida, 2013). VD is a process whereby wet material (fresh sample) is dried under sub-atmospheric pressure. During the vacuum drying processing, the samples are place on a heating panel in the drying chamber, then the air is expelled from the drying chamber to a constant vacuum pressure state (Xie et al., 2018). During VD, water molecules travels to the surface from inner and evaporate to vacuum chamber. Vapor pressure gradient is generated when the partial vacuum in the drying chamber reduce water vapor concentration at the product surface by conduction (Dev and Raghavan, 2012; Karam et al., 2016; Parikh, 2015). This is due to the reduced pressures which expand and remove the water molecules into the vapor phase (Dev & Raghavan, 2012). Thus, vacuum drying is able to evaporate water at lower pressure compared to higher ones (Perazzini et al., 2015). The heat is transferred by conduction and the product temperature can be controlled easily (Lewicki, 2006). In the food industry, vacuum oven drying is generally carried out together with some other drying methods such as vacuum freeze drying and microwave freeze drying. The pressure and temperature used in previous studies are shown in Table 18.

Table 18 Condition of vacuum oven drying on citrus fruits based on previous studies

Sample	Pressure level (mbar)	Temperature (°C)	Reference
Lime residues	100	60, 70, 80	Jongaroontaprangsee et al. (2014)
Lemon slices	800	60, 65, 70, 75	Wang et al. (2018)

2.5 Effects of different drying treatment on physico-chemical properties

Physicochemical properties are easily affected by the drying process. Different approaches of drying process may influence the physicochemical characteristic at a different levels. Previous studies have shown that physical properties such as color, water activity, vitamin C, and pH were simply changed after being dried. Table 19 shows

the influence of different drying treatments on physicochemical properties of citrus fruits. Based on Table 19, changes of vitamin C and caretenoids compound have been significantly affected by hot air drying methods (Marey and Shoughy, 2016), however freeze drying has shown minimal changes to the pomelo peels (Abd Rahman et al., 2016). Similar observation was observed in terms of color for microwave drying of mandarin citrus peels done in a study by Ghanem et al. (2012). In contrast with hot air drying methods, the color was significantly changes during elevated temperature (Nesrine et al., 2015). In addition, vitamin C content was highly observed in freeze dried citrus "Hallabong" powder compared to hot air dried (Lee et al., 2012).

Table 19 The influence of different drying treatment on physicochemical properties of citrus fruits**.**

Sample	Drying Operation	Significant findings	Reference
Citrus peels (Mandarin and orange peels)	Laboratory-scale hot-air dryer at air temperatures from 40 to 70 °C under a constant air velocity of 1 m/s.	● Over-drying CPs with the higher temperatures and to a final moisture level of 5.4 ± 0.2% sharply increased the loss of vitamin C, carotenoids as antioxidants and essential oils.	Marey and Shoughy (2016)
Lemon peel	Air drying temperature (40, 50 and 60°C).	● L* and b* decreased (indicating lower luminosity and lower yellowness, respectively) and later increased (indicating higher redness) with increasing drying temperature.	Nesrine et al (2015)
Pomelo peel	Freeze drying (FD) Samples were quenched in liquid nitrogen and subsequently vacuum freeze-dried overnight in a freeze-dryer at 2 °C and 1.25 mbar	● Freeze-drying markedly reduces the relative energy dissipation capacity of the peel. ● Measuring the transmitted force during impact furthermore indicated a transition from a uniform collapse of the foam-like tissue to a progressive collapse due to water extraction ● Maxwell model illustrates that freeze-drying not only drastically reduces the	Thielen et al. (2015)

Sample	Drying Operation	Significant findings	Reference
		damping function of the dashpots but also stiffens the springs of the model.	
Citrus “Hallabong” powders	Hot air (60±1 ℃ for 96 hr) and freeze drying (frozen by a deep freezer at −70 ±1℃ for 24 h and then dried for 72 hr by a freeze dryer).	• Vitamin C was high in freeze-dried whole fruit powders (220.8-364.7 mg / 100 g) compared with those in hot air-dried ones (80.1-114.6 mg / 100 g). • Browning index of freeze-dried powders was significantly lower than those of hot air-dried ones. • Bulk densities, compaction densities, and Hausner ratios of the powders were significantly higher in freeze drying method compared with hot air drying method. • Water solubility and hygroscopic behavior of freeze-dried powders were higher than those of hot air-dried ones. • In conclusion, ‘Hallabong’ powders can be made using freeze drying method with high quality in terms of vitamin C content, color, and water solubility.	Lee et al. (2012)

2.5.1 Effects of different drying methods on total phenolic content

Processing treatment, as well as environmental factors such as temperature and light intensity exposure, could also affect the phenolic contents (Nayak et al., 2015). Furthermore, the differences or variation in plant cultivar as influenced by genetic factors, maturity stage, cultural practices and post-harvest conditions also could affect the quantity and quality of the phenolic compounds (Odriozola-Serrano, et al., 2008; Pichaiyongvongdee et al., 2014; Zefang et al., 2016). Interestingly, according to Abdullah et al. (2012), the natural active ingredients in fruits were found in their peels and decreased in concentration towards the flesh or pulp. However, the changes of the total phenolic composition also can be occurred based on drying operation applied. Table 20 shows the effects of different drying treatments on the total phenolic content of citrus fruits selected. The table illustrates the drying temperature does affect the total phenolic content for instance, TPC showed a decrease trend when drying temperature increased in study done by Jongaroontaprangsee et al. (2014) which could be due to the decomposition of antioxidant components after being exposed to thermal process (Senevirathne et al., 2009). In contrast to a study by Chen et al. (2011) which they found that, during elevated temperature, the TPC increased significantly, this is might be because of the occurrence of nonenzymatic reaction such as Maillard reaction and caramelisation when high temperature involved could be the reason of high TPC content (Nicoli et al., 1999; Liu et al., 2007; Chen et al., 2009).

2.5.2 Effects of different drying methods on antioxidant capacity.

Apparently, as drying process applied to preserve and extend the shelf life of a product, the quality and the bioactive compound exhibited antioxidant capacity will be affected simultaneously. However, the information regarding the influence of drying methods on nutritional and bioactive compound of citrus fruits is scarce. Recently, great attention was discovered as heat treatment and their effect on enhancing bioactive phytochemical content with antioxidant activities. Therefore, the effects of different drying methods on antioxidant of citrus fruit are summarized in Table 21. The

results of antioxidant capacity vary between the types of citrus, origin, different part of tissues and drying methods involved during drying process.

Zou et al. (2016) reported that the compound of the phytochemical profile could be varied based on different species and cultivars that contributed to different biological properties. However, different drying temperatures also showed different effects on the FRAP values. Briefly, the results of the current study showed a considerable variation of antioxidant activity among different fruits (Sultana et al., 2012).

Table 20 Summary of the effects of different drying methods on total phenolic content of citrus fruits.

Citrus fruits	Drying process	General by-products	Pulp	References
Pomelo from Vietnam	Hot air drying; 50 °C; 24 hr	4.08 - 7.50	NA	Hung et al. (2020)
Citrus *unshiu* peels	Pre treatment – 60 °C for 48 hr; Treamtent : roasted at 100, 120, 140, 145, 150,155 °C	28.50 - 48.20	NA	Ko et al. (2020)
White Tambun Pomelo	Freeze drying (FD)	105.75*	NA	Lim and Loh (2016)
Kaffir Lime		74.22*	NA	
Lime		117.78*	NA	
Calamansi		100.92*	NA	
Limes (*Citrus aurantifolia* Swingle)	Fresh	1021.42*	NA	Jongaroontaprangsee et al. (2014)
	Pretreatment (blanching only)	894.82*	NA	
	Pretreatment (blanching+ soak in ethanol)	837.49*	NA	
	Residues after hot air drying at 60°C	751.85*	NA	

Citrus fruits	Drying process	General by-products	Pulp	References
	Residues after vacuum drying		NA	
	60°C	810.74 #	NA	
	70°C	812.01 #	NA	
	80°C	802.98#	NA	
	residues after LPSSD at		NA	
	60°C	808.16 #	NA	
	70°C	808.03 #	NA	
	80°C	801.80#	NA	
Pomelo (Ledang)	Freeze drying (FD)	30.66 – 63.11 mg/g FW	NA	Chang and Azrina (2017)
Gallega "lime fruits"	Freeze drying (FD)	42.76*	NA	Esparza-Martínez et al. (2016)
	Tray dryer			
	60°C	37.26	NA	
	90°C	37.77	NA	
	120°C	63.28	NA	
Pomelo Tambun	Fresh Juice extracted	NA	385.3 mg GAE/L	Shah (2015)
	Juice extracted treated with enzyme	NA	368.2	
Pomelo Ledang	Fresh Juice extracted	NA	497.7	
	Juice extracted treated with enzyme	NA	467.7	
Tambun White	Freeze drying (FD)	406.65	70.56	Toh et al. (2013)
Tambun Pink		300.56	61.72	
Pomelo from China	Hot air drying at 50°C	53.64*	NA	Ding et al. (2013)
'Liangpinyou' (LP)				
'Duanshiyou' (DS)		46.02	NA	

Citrus fruits	Drying process	General by-products	Pulp	References
‘wubuyou’ (WB)		42.79	NA	
‘beibeiyou’(BB)		46.06	NA	
Pomelo from Thailand	Freeze drying (FD)			Mäkynen et al. (2013)
Kao-Yai (KY)		NA	113.73*	
(Thong-Dee) TD		NA	101.32	
Kao-Tangkwa (KT)		NA	102.57	
Kao Numpueng (KN)		NA	115.02	
Ta-Koi (TK)		NA	110.52	
Tubtim Siam (TS)		NA	107.23	
Pomelo from Thailand	Freeze drying (FD)	1.10-2.16 * (Flavedo)	NA	Pichaiyongvongdee et al. (2014)
		1.18-3.38 (Albedo)	NA	
		3.11-4.96 (Segment membrane)	NA	
		0.83-2.27 (seeds)	NA	

Note: NA: not available

* Total phenolic content of pomelo was expressed as mg gallic acid equivalent/g dry weight of extract

Total phenolic content of pomelo was expressed as mg GAE/g dry basis

Table 21 Effects of different drying methods on antioxidant capacity of citrus fruits.

Sample	Treatment	Parts of fruits	DPPH (%)	FRAP (mM Fe2+/g FW)	References
Pomelo from Vietnam					
Da Xanh	Tray drying, 50 °C	Peels	23.60 -32.30	NA	Hung et al. (2020)
Nam Roi			13.70 – 24.00	NA	
Tan Trieu			34.80 – 43.30	NA	
Citrus unshiu	Roasted at 140 °C		756.23*	NA	
	145 °C	Peels	648.34*	NA	Ko et al. (2020)
	150 °C		613.86*	NA	
	155 °C		555.61*	NA	
Lemon	Fresh		1.3*	0.38	
	Freeze drying (FD)		0.82*	0.51	
Key lime	Fresh	Peels	1.83*	0.44	Azman et al. (2019)
	Freeze drying (FD)		1.57*	0.47	
Musk lime	Fresh		3.16*	0.46	
	Freeze drying (FD)		2.7*	0.53	
Pomelo Ledang Variety	Freeze drying (FD)	Flavedo	166.30 (µg/ml)	307.59	Chang and Azrina (2017)
		Albedo	168.00	245.88	
		Lamella	167.45	136.84	
Gallega Lime wastes	Freeze drying (FD)	Lime wastes (extractables)	247.59	NA	Esparza-Martinez et al. (2016)
	Tray drying				
	60℃		122.28	NA	
	90℃		175.61	NA	
	120℃		294.92	NA	

Table 21 (Continued)

Sample	Treatment	Parts of fruits	DPPH (%)	FRAP (mM Fe2+/g FW)	References
Gallega	Freeze drying (FD)		683.54	NA	Esparza-Martinez et al.
Lime wastes	Tray drying			NA	(2016)
	60°C		671.95	NA	
	90°C	Lime wastes	672.97	NA	
	120°C	(non-extractables)	659.68	NA	
Tambun White		Peel	NA	1.01 mmol Fe (II)/100g FW	Toh et al. (2013)
	Freeze drying (FD)	Pulp	NA	0.6	
Tambun Pink		Peel	NA	0.65	
		Pulp	NA	0.51	
	Freeze drying (FD)		37.30	NA	
	oven drying				
	50°C		38.30	NA	
Oranges from Yunlin, Taiwan	60°C	Peels	29.10	NA	Liu and Tsai (2012)
	70°C		46.20	NA	
	80°C		68.40	NA	
	90°C		70.20	NA	
	100°C		80.10	NA	
Pomelo from Bangkok, Thailand	Freeze drying (FD)	Pulp	NA		Mäkynen et al. (2013)
Kao-Yai (KY)				443.56	
Thong-dee (TD)			NA	345.78	
Kao-Tangkwa (KT)			NA	395.22	
Kao-Numpueng (KN)			NA	616.89	
Ta-Koi (TK)			NA	386.33	
Tubtim Siam (TS)			NA	377.44	

Note: NA: not available. * The data reflects the Inhibition Concentration (IC) or EC_{50} in mg/mL.

2.6 Mathematical Modeling

The mathematical model can be used to describe the convective drying process. Hashim et al. (2014) explained the significance of the process design and quality control, which includes the knowledge of temperature and moisture distribution. Numerous studies about modeling used to describe drying process; however, there is still lack of information on drying kinetics of the citrus product. The models help to design more efficient conventional drying (convective hot air dryers) by reducing time loss during drying and improving the drying process of the product. Furthermore, an equation obtained from the mathematical modeling of the drying process is capable to be used as a reference to select the most suitable operating conditions (Onwude et al., 2016a).

Therefore, exploration of the thin layer modeling approach as an essential tool in drying kinetic estimation from experimental data, drying behavior description, refining the drying process and could be used to reducing the total energy treatment. Using the mathematical modeling, it involves an exponential trend line and forces the intercept to be equal to the initial dry basis of the product (Onwude et al., 2016b) which provided by Equation 2.1.

$$MR = M_o \exp^{-kt} \quad (2.1)$$

Where *MR* is the moisture ratio; *M* is the wet basis of moisture content at any time, (unit); M_o is the initial wet basis moisture (unit); M_e is the equilibrium moisture content (unit).

Moisture ratio was calculated according to Equation 2.2 to compare the drying of product with different initial moisture contents based on the wet basis moisture at any time, *t*.

$$MR = \frac{M - M_e}{M_o - M_e} \quad (2.2)$$

However, the values of M_e are relatively small in comparison M and M_o, thus the error interrelates in the simplification is negligible. Therefore, Equation 2.2 becomes Equation 2.3.

$$MR = \frac{M}{M_o} \tag{2.3}$$

Through this approach, the exponential trend line described in Equation 2.3 becomes Equation 2.4.

$$MR = \frac{M}{M_o} = exp^{-kt} \tag{2.4}$$

According to Torki-Harchegani et al. (2016), the pressure effect is negligible compared to the temperature and moisture effect. Several models have been widely used in drying process which can be referred in Table 22.

Table 22 Mathematical models applied to drying curve

Model name	Model expression	Reference
Wang and Singh	MR= 1+ at+bt^2	Wang and Singh (1978)
Newton	MR= exp(-kt)	Ayensu (1997)
Henderson and Pabis	MR= a exp(-kt)	Chhinman (1984)
Logarithmic	MR= a exp(-kt) + b	Yaldiz et al. (2001)
Midilli and Kucuk	MR= a exp(-kt^n) + bt	Midilli et al. (2002)
Two-term exponential	MR= a exp(-k_1t) +b exp (-k_2t)	Henderson (1974)
Diffusion approach	MR= Iexp(-kt) + (1-a)exp (-kbt)	Kassem (1998)

Source: Torki-Harchegani et al. (2016)

As drying involves complex simultaneous heat and mass transfer, food drying is relatively more complicated because of the different resources leading to a

significant variation of composition and physical structure of the food constituents (Erbay and Icier, 2010; Pereira et al., 2015; Talens et al., 2016). Therefore, effective mathematical models validated using experimental studies can be used for operation in drying purposes such as design, simulation, optimization, energy integration, and the controller (Kesbi et al., 2016; Van Boekel, 2008). The selected models can be used to design a related dryer and improved the drying process of the product by observing the weight loss and reduce the drying time.

Mathematical models can be categorized into 3 known types, namely: theoretical, semi-theoretical and empirical. Theoretical models commonly applied based on an assumption which might not relate or explain the actual drying operation. Meanwhile, empirical and semi-theoretical models are always applied in the drying of fruits, vegetables, and other crops by using mathematical models of thin layer drying which is more practical and provide relevant results (Erbay & Icier, 2010; Kesbi et al., 2016). Numerous studies on mathematical models have been done on citrus fruits such as lemon slices (Darvishi et al., 2014; Sadeghi et al., 2013; Torki-Harchegani et al., 2016; Wang et al., 2018), lemon peels (Nesrine et al., 2015) and grapefruit seed (Cantu-Lozano et al., 2013) which can be refer in Table 23.

Table 23 Modeling approach of previous study related to citrus fruits

Agricultural crops	Drying Mode	Drying process conditions	Significant findings	Modelling approach	References
Pomelo peel	Freeze drying	T = -30 °C	The minimum and maximum Deff value was 2.026×10^{-9} – 8.106×10^{-9}	Diffusion and Henderson-Pabis model	Tuncer et al. (2020)
	Microwave drying	Power = 350 W	The minimum and maximum Deff value was 1.925×10^{-8} – 7.295×10^{-8}	Logarithmic model	
	Forced convection drying	T = 90 °C	The minimum and maximum Deff value was 1.418×10^{-8} – 5.674×10^{-8}	Logarithmic model	
Pomelo albedo	Freeze drying (FD)	Cold trap temperature: −80/−55 °C, vacuum degree: ≤10 Pa	Drying time of samples were achieved as 24 min, 34 min, 410 min for 5×1×0.5 cm-sized samples and 30 min, 44 min and 540 min for 5×1×1 cm-sized samples for MWD, FCD, and FD, respectively	artificial neural network (ANN)	Kırbaş et al., (2019)
	Microwave drying (MWD)	Power = 350W T = 90 °C			
	Forced convection drying (FCD)	T = 90 °C			
Lemon slices	Pulsed vacuum drying (PVD)	T = 60, 65, 70, and 75 °C	The minimum and maximum Deff value was 1.66×10^{-11} and 1.90×10^{-10} $m^2 s^{-1}$ at drying temperature of 60 and 75 °C, respectively	Weibull model (exponential model)	Wang et al. (2018)

Table 23 *(Continued)*

Agricultural crops	Drying Mode	Drying process conditions	Significant findings	Modelling approach	References
Lemon slices	Infrared-vacuum drying	h =0.5 cm; d = 4 cm ;Infrared Radiation power =300, 350, and 400 W; Pressure = 5, 15, and 25 kPa; t = 0–140 min	Values for the effective moisture diffusivity of lemon samples were obtained in the range of 2.92×10^{-10} – 1.58×10^{-9} m^2/s.	Quadratic model	Salehi and Kashaninejad (2018)
Lemon	Microwave hot air drying (MW-HAD)	h = 5 mm; d = 5mm; T = 50 – 60 °C; P = 185.5 and 388.5 W;(specific P = 0.97 and 2.04 W/g)	Reduced drying time of about 20-30 when compared with using HAD alone	Semi theoretical Midilli et al. model	Kesbi et al. (2016)
Lemon slices	Hot air drying (HAD)	T = 50, 60, 75 °C	Effective moisture diffusivity was determined on the basis of Fick's second law and obtained to be 1.62×10^{-11}, 3.25×10^{-11} and 8.11×10^{-11} $m^2\ s^{-1}$	Midilli and Kucuk model	Torki-Harchegani et al. (2016)
Citrus lemon (*Citrus limon* L.)	Micowave (MW)-Hot Air Drying (HAD)	h = 5 ± 1 mm; v = 1m/s; T = 22 °C; P = 180-720 W	-	Semi theoretical Midilli et al. model	Darvishi et al (2014)
Lemon slices	MW-HAD	h =5 mm; d = 50 ± 3 mm; T = 50-60 °C; R.H = 20-95%; P = 185.5 and 388.5 W; (Specific P= 0.97 and 2.04 W/g respectively)	MW-HAD = 17-31 times reduction in drying time; D_{eff} increased 21-38 times as compared to HAD; D_{eff} = 4.116×10^{-9}-7.618×10^{-10} m^2/s	Diffusion models (Dincer and Dost; Crank models)	Sadeghi et al. (2013)
Grapefruit seeds	HAD	T = 40, 50, 60, and 70°C; v = 0.6, 1.0 and 1.4 ms^{-1}	Effective moisture diffusivity in grapefruit seeds ranged from 4.36×10^{-10} to 6.82×10^{-10} m^2s^{-1}.	Page model	Cantu-Lozano et al. (2013)

2.6.1 Degradation constant of kinetic modeling

Normally, industry sectors have to guarantee a minimal food quality loss throughout storage condition. Under those conditions, the storage duration and pre-treated drying condition are important variables to be monitored (Touati et al., 2016). For this, degradation kinetic modelling is a significant step to control and predict quality parameter changes during processing and storage (Remini et al., 2015). In addition, the empirical approach of the kinetic modelling based on the reaction order (corresponding to the zero, half-, first- and second- order reaction modelling) can be used to predict shelf life (Ling et al., 2015; Van Boekel, 2008). The Arrhenius equation has been extensively applied to quantify the effects of storage time on the rate of several biochemical reaction. Significant knowledge of the kinetic parameter is essential to predict the change antioxidant quality that happen during storage (Kim et al., 2018). Thus, data on kinetics model can be useful in determining the processing of pomelo albedo's extract. Kinetic degradation studies on different drying temperatures have been reported in citrus juices (Igual et al., 2011; Remini et al., 2015; Touati et al., 2016). No recent research available in the literature on degradation kinetic parameters of TPC, naringin and antioxidant capacities from pomelo residues extract during storage. Nevertheless, previous research on kinetic modeling of citrus related product during storage were used as references to carried out the storage analysis were summarized in Table 24.

2.7 Effects of storage time on phenolic composition and antioxidant capacities

Fascinatingly, citrus peels has been proven containing phenolic compound namely naringin that that plays important role in human health by their anti-oxidative effects and capable to lowering the risk of heart diseases (Castro-Vazquez et al., 2016; Lv et al., 2015; Rafiq et al., 2019; Wadhwa and Bakshi, 2013). For this reason, citrus fruits famously known as 'health promoting fruit', with high commercial value and increasing production worldwide. Nevertheless, short shelf life is the main challenge due to rapid softening and skin wrinkling of fresh citrus fruits due to water loss and

fruit decay. Most of the previous study have reported on storage for instance, the effects of storage time on quality (ascorbic acid, phytochemical; eg. phenolic, and flavonoid composition; neohesperidin, naringin, naringenin) based on extraction of citrus juice such as blood orange juice (Remini et al., 2015; Touati et al., 2016; Wibowo et al., 2015), grapefruits juice (Shah, 2015; Agudelo et al., 2017; Igual et al., 2010; Moraga et al., 2012) were discovered. The corresponding research relates well as juice extraction processing were commonly applied and have high value in industrial scale, especially for citrus fruits comparing with the residues parts. The storage analysis is critically significant to be carry out in order to identify the expired date and the shelf life of a product.

In addition, prior to the juice extraction process, the shelf-life of citrus fruits (after post-harvest) need to be investigated to identify the limit of acceptability in commercial sectors. By observing the quality changes occurred at certain period, the condition of storage such as exposure of oxygen, temperature, relative humidity and other significant factors need to be to improve and maintain the shelf- life commodities. Longer period of storage time is preferable as it can be stored longer while maintaining the quality and chemical composition, then, also can be exported to other countries that need long hour to be transported. Therefore, physicochemical were recorded following storage on fresh citrus fruits (Chaudhary et al., 2017; Chebrolu et al., 2012; Salihah, 2015; Sirisomboon and Lapchareonsuk, 2012), the quality involved such as to changes of weight loss, color, size, chemical composition, throughout storage time.

In general, the research on shelf life of fruits exploration basically focusing on juice and fresh fruits instead of the fruit's residues. Nevertheless, recent discovery of potential health compound were observed in fruits residues (Nesrine et al., 2015; Romdhane et al., 2016; Toh et al., 2013) and their effect of processing (i.e. drying) on the quality of targeted peels/residues were reported (Table 24) during storage time. Nevertheless, limited information available regarding the effects of storage time on bioactive compound and antioxidant capacities of vacuum dried extracts of pomelo residues (albedo).

Table 24 Previous study on storage analysis of citrus related products

Raw material	Treatment	Significant Findings	References
Rio Red grapefruit	Freeze drying 12 weeks at 11 and 5° C, Control conditioned at 16°C for 7 days before stored up to 6 days.	• FD pulp from irradiated fruits had a higher flavonoid content (naringin and narirutin) than freeze dried and control. FD reduced the lycopene content, but the reduction in B carotene occurred only in the control fruit. • Reduction in d-limonene and myrcene was observed in the irradiated fruits at 6 days after harvest and FD samples.	Chaudhary et al. (2017)
Lemon juices	Pasteurized at 90 °C for 15 sec and stored for -25 °C for 180 days	• Fifteen phenolic compounds were determined in the lemon juice and the most abounded phenolic compounds were hesperidin, eriocitrin, chlorogenic acid and neoeriocitrin. • In generally, phenolic compound concentrations of lemon juice samples increased after the pasteurization treatment. • Four carotenoid compounds (β-carotene, β-cryptoxanthin, lutein and zeaxanthin) were detected in natural cloudy lemon juice. • Lutein and β-cryptoxanthin were the most abounded carotenoid compounds in the lemon juice. • Color values of the lemon juices were not affected by processing and storage periods. • HMF and browning index of the lemon juices increased with concentration and storage. • According to the results, storing at −25 °C was considered as sufficient for acceptable quality limits ofnatural cloudy lemon juice	Uçan et al (2016)

.Table 24 (Continued)

Raw material	Treatment	Significant Findings	References
Orange peel powder in ghee	Hot air oven attached with blower at 40 °C for 8 hr were applied Different storage temperatures: (T_1:6±2 °C; T_2: 32±2 °C; T_3:60±2 °C); storage period of 21 days. Different treatment: solvent to sample ratio (10:1, 13.5:1 and 17:1, Ml/g), extraction temperature (30, 40 and 50 °C) extraction time (6, 12 and 24 h)	• PV, TBA and FFA of ghee samples increased significantly while radical scavenging activity (RSA) of ghee samples decreased significantly at accelerated temperature (T_3) as compared to the temperatures at T_1 and T_2. • Effect of storage temperature on development of peroxides and TBA of ghee samples was significantly higher than the effect of treatment and storage period while treatment had more significant effect on the change in FFA and RSA as compared to storage temperature and storage period • Ghee incorporated with orange peel extract (OPE) showed stronger activity in quenching DPPH radicals and least development of PV, TBA and FFA than ghee incorporated with BHA and control.	Asha et al. (2015)

CHAPTER 3
EXPERIMENTAL DESIGN AND METHODOLOGY

The study was segregated into three stages as shown in Figure 5. The activities that were carried out addressed the aims of this study; which were the effect of different drying methods (freeze drying, conventional hot air oven and vacuum oven) on the physicochemical properties (color, nutritional composition) and antioxidant capacity between different parts of pomelo residues (flavedo, albedo, lamella and pulp waste); selection of pomelo residues and ideal drying condition of selected pomelo residue were identified; to identify the best mathematical model of drying method and drying temperature (50, 60, 70, 80 and 90 °C) and to fit drying experimental data into the suitable model at selected parts of pomelo residues and finally, determination of major phenolic compounds (naringin) were carried out using HPLC and antioxidant capacity (radical scavenging activity and ferric reducing power) of selected dried pomelo residues extract during storage at 8 °C for 12 weeks. In section 3. 1, different parts of pomelo residues were manually peeled and undergoing a proposed drying process at different drying temperature. Meanwhile, the time of the drying process was controlled. The evaluation of the best drying method was carried out and the optimal quality retention of pomelo residues was determined based on the criteria as elaborated in section 3.8. The study was further extended by determining the best fit model for drying kinetics at the best drying temperature and drying method in section 3. 9. The last part of the project, as explained in section 3. 11, involved the determination of phenolic profile and the effects of proposed storage period on kinetic models based on antioxidant capacity and phenolic composition of pomelo residues.

3.1 Research design

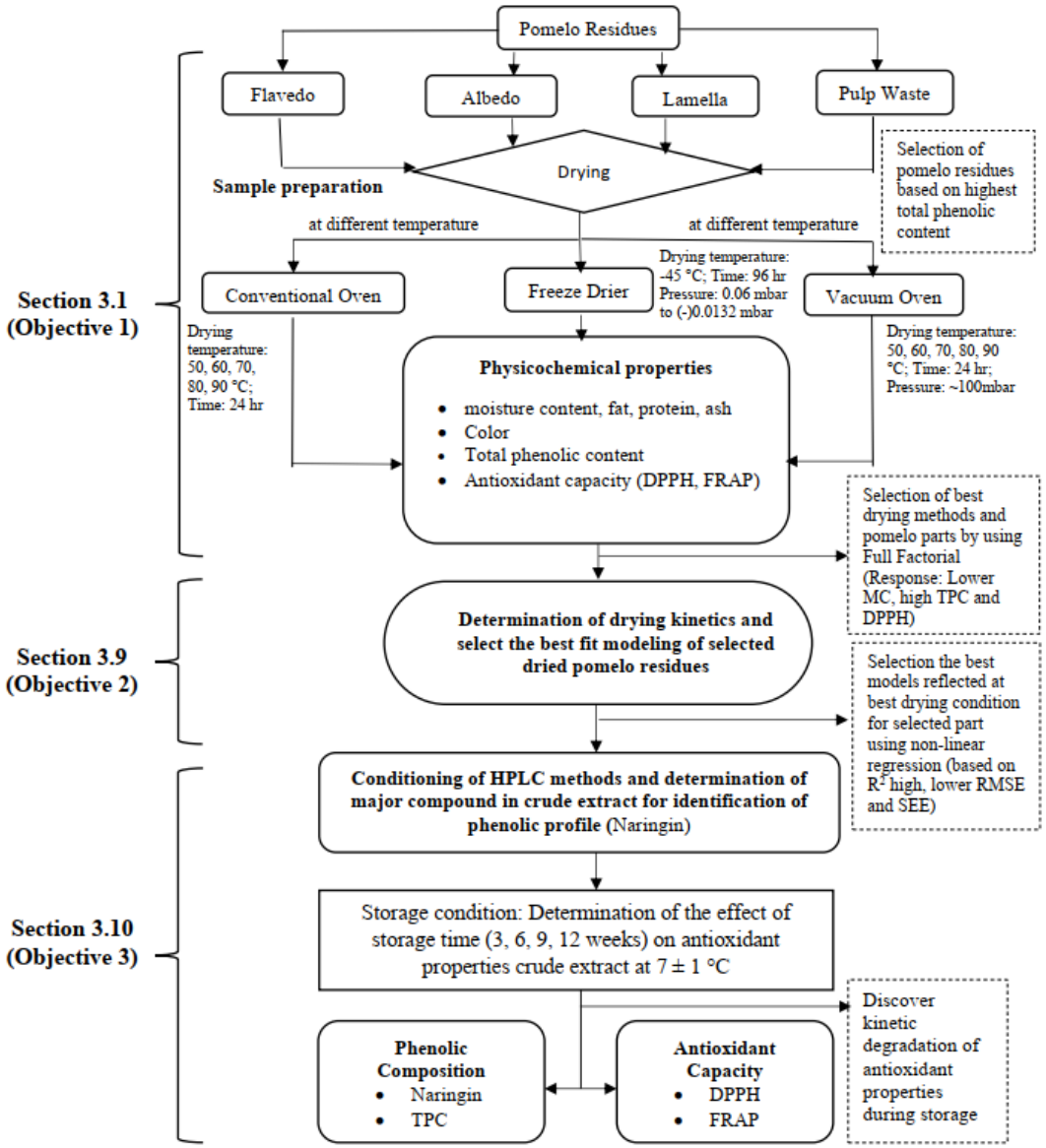

Figure 5 Research design

3.2 Preparation of pomelo fruits.

Pomelo fruit from variety Tambun White (*Citrus grandis*) PO52 (Figure 6) was used in this study. It is the most cultivated and popular pomelo variety among consumers in Malaysia. Fruits were harvested at commercial maturity stages which were taken from the commercial orchard in Department of Agriculture Kinta District,

Ipoh, Perak, Malaysia in April 2015. The fruits were selected based on uniformity of size, color, and commercial maturity stage. Harvesting season was conducted based on a definite period of time and it was started counted after the full bloom stages. The first harvest started when the fruits were 6 months into growth after full bloom and judge as immature. Early mature fruits were harvested at 6 ½ months and late mature fruits at 7 months. At 7 ½ months after full bloom, the fruits were harvested as over-mature (Terdwongworakul et al., 2009).

Figure 6 Harvested Tambun White (*Citrus grandis*) PO52 fruits.
Source: Sirisomboon and Theamprateep (2012)

The fruits were washed to remove soil and contaminants, then manually peeled and separated from the 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 can be seen in cross section of pomelo fruit in Figure 7.

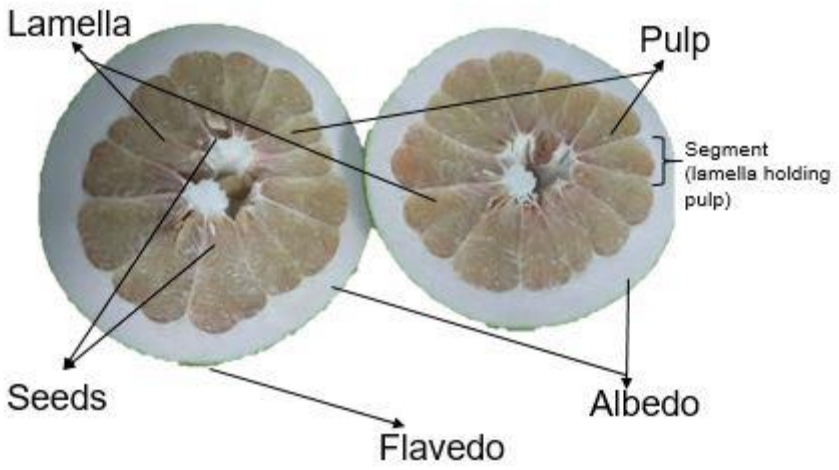

Figure 7 Cross section of pomelo fruit

The albedo, flavedo and lamella were cut into cubes of approximately 1 cm^3, 1.0 cm x 1.0 cm x 0.3 cm and lamella 0.5 - 1 cm x 1 - 1.5 cm x 0.1 - 0.5 cm, respectively. The consistent shape or size was used to make sure the distribution of heat treatment during drying was consistent between the products. The dimension or size between the parts of pomelo peels was considered different due to the naturally existed of the pomelo peels, which can be seen in Figure 8. In addition, the pulp was pressed manually with a fruit press juicer (CL-003AP, Interglobal International, LTD, Taiwan) according to Salihah et al. (2015). The waste pulp and the juice were collected separately, besides, for the pulp waste, it was place by spreading out with the 0.2cm thin layer of thickness (Shamsudin et al., 2015) on a flat tray before drying take place. The seeds were not separated with the pulp, and it could be included with the pulp waste after pomelo juice was extracted. The weight (kg) of each pomelo residues were recorded prior to drying treatment.

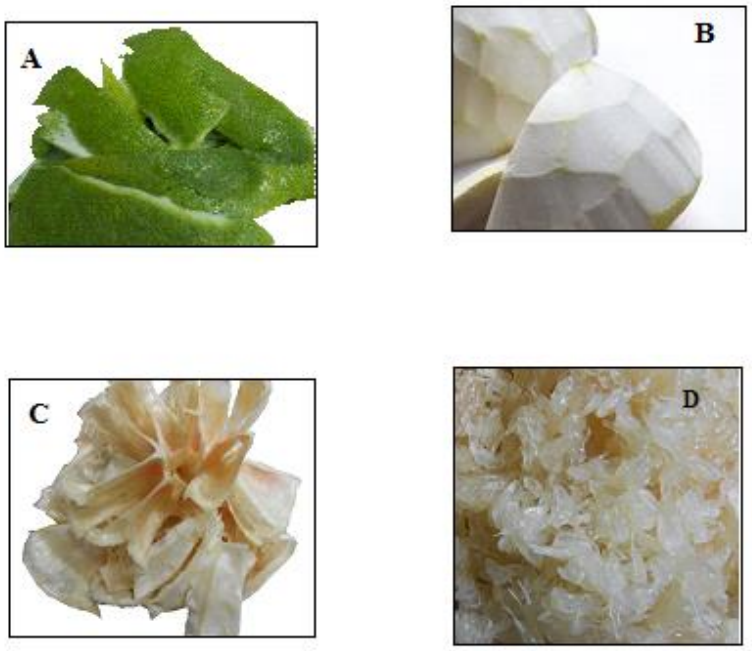

Figure 8 Parts of pomelo residues (A) Flavedo; (B) Albedo; (C) Lamella; (D) Pomelo pulp.

The samples were then be stored in a freezer (HVF-301S; Hesstar Corporation Sdn. Bhd., Shah Alam, Malaysia) located in F2.2.A (Food Processing Machinery Design) laboratory, at -18 ˚C to maintain their freshness.

3.3 Drying Methods

The three different methods of dehydration used were convection oven drying using a DO6836 oven (Memmert GmbH, Schwabach, Germany), freeze drying using a Benchtop K device (SP Scientific, Gardiner, NY, USA) and vacuum oven drying (VD23, Binder GmbH, Germany).

3.3.1 Conventional oven drying

Conventional drying comprises the additional of heated air at atmospheric pressure and releases water vapour from the foodstuff (Perazzini et al., 2015). Firstly, 100 g of fresh pomelo residues (albedo, flavedo, lamella, pulp waste) were spread as a single layer on a separate tray. The drying method was carried out at Biomaterials

Engineering Properties and Nutraceuticals laboratory, Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia (UPM). The pomelo residues were subjected to dehydrate using convection DO6836 oven (Memmert GmbH, Schwabach, Germany) at 50, 60, 70, 80, 90 °C (Chen et al., 2011) for 24 hr to be completely dried. The choice of temperature was made according to Garau et al. (2007) based on hot air drying for orange peels . Dried orange skin showed quite resistant to the different drying temperature (40 - 70 °C), however, major modification was observed on dietary fibre compound when either extended drying period at lower or elevated drying temperature were conducted (Garau et al., 2007). Nevertheless, lower temperature needs a longer time to dry the product in comparison to higher temperature (Marey & Shoughy, 2016). Thus, by using final moisture content (should be less than 10 %) as a reference, over-drying, reduction of quality, and antioxidant capacity of pomelo residues could be prevented (Marey & Shoughy, 2016). The dried sample was finely ground into powder form using grinder (SM200, Rostfrei, Germany) after being dried to final moisture content lower than 0.1 kg/kg dry basis (Nesrine et al., 2015).

3.3.2 Freeze drying

Freeze drying is a sublimation process of water in solid (ice) form from a frozen material (Geankoplis, 2003) into vapor form. The freeze drying process was used as a control in this study as it has been commonly known as the most effective method for retaining product characteristics in comparison with other drying process (Perazzini et al., 2015; Azman et al., 2019; Karam et al., 2016; Karaman et al., 2014; Strummilo & Adamiec, 1996; Vanamala et al., 2005; Chen et al., 2011). The reason of "untreated' sample not being selected in this study because of fresh fruits are highly perishable commodities (due to their high moisture content, around 80 percent (refer Table 4.2) that deteriorate within a short period of time if treated improperly (Orsat et al., 2006; Sirisomboon et al., 2012). The reaction of the microorganism favors this condition and may simultaneously spoil the fresh pomelo residues. Thus, drying process reduce the free moisture availability for microorganism reaction in dried matter form. In addition, the yields of extraction for fresh citrus is 3-5 times lower than freeze dried citrus based on Azman et al. (2019) which confirms the freezing condition is an effective method

to preserve the maximal yield of bioactive compound. Freeze drying were conducted at Nutrition Laboratory 1, Department of Nutrition and Dietetics, Faculty of Medicine and Health Science, UPM. 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 hr at the range of 0.0600 mbar to (-)0.0132 mbar (Benchtop K; SP Scientific) until they were completely dried. This is the optimal condition (temperature and time) (Dzung, 2012) to fully frozen the samples in order for the temperature of product to reach the optimal freezing temperature and change to the solid phase. Then, sublimation process occurs from solid to vapor phase in a vacuum drying stage by evaporation (Dzung, 2012).

3.3.3 Vacuum oven drying

Vacuum oven drying involves the evaporation of water at lower pressure and the heat was indirectly added by contact with a metal wall or by radiation (Perazzini et al., 2015). Vacuum drying was conducted in a vacuum oven proposed by Devahastin et al. (2004) at Food Processing Quality Laboratory, Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia (UPM). Vacuum drying operation was conducted at 50, 60, 70, 80, 90 °C at an absolute pressure of 95 - 105 mbar (Bradley, 2010) for 24 hr. The temperature and drying time (24 hr) were selected based on similar application on conventional drying. Approximately 100 g of the prepared residues were placed as a thin layer with a thickness 0.2 cm on an aluminum foil (21 cm x 21 cm) to be dried in vacuum oven (VD23, Binder GmbH, Germany).

3.4 Preparation of powder from dried pomelo residues

The dried sample was eventually ground into fine powder using a grinder (Waring, Torrington, CT, USA) and sieved using a sieve analyzer (Retsch, Haan, Germany) to obtain a particle size 1 mm. Subsequently, it was sieved using a 1 mm siever analyzer (Retsch, Haan, Germany). Later, the dried sample was stored at -18 °C in a freezer (HVF-301S; Hesstar, Malaysia) before further analysis. Grinding and sieving were conducted to obtain small and homogenous particle size of pomelo peel powder. Small particle size increases the surface area of extraction and eventually boosts the

rate of extraction yield (Azmir et al., 2013). This dried powder was used for physicochemical properties and antioxidant analysis.

3.5 Physiochemical properties

Physicochemical properties include the color, proximate analysis, total phenolic content, and antioxidant capacities of pomelo residues.

3.5.1 Color analysis

The color of pomelo fruit is very important for their commercial acceptability. The color of pomelo peel was measured using colorimeter (CR-10; Konica Minolta, Osaka, Japan) (Alibaş, 2012). The color measured based on lightness (L*) with the range of 0 (blackness) and 100 (whiteness). According to the International Color Standardisation body in terms of Commission Internationale de L'Eclairage (CIE) - lightness (L*) is the light to dark spectrum; redness or greenness (a*) is red to green spectrum; and blueness or yellowness (b*) is blue to yellow spectrum. The color of the samples was measured at three different points of the samples. Differences of L*, a*, b* were calculated using the Equation 3.1, 3.2 and 3.3 and were used to determine the change in different color analysis.

$$\Delta L* = L* - L_o* \quad (3.1)$$

$$\Delta a^* = a^* - a_0^* \quad (3.2)$$

$$\Delta b^* = b^* - b_0^* \quad (3.3)$$

The results of fresh pomelo residues were tabulated in Table B1 (Appendix B) for references.

Chroma indicates the purity of the color which refers to red-green (+/-a*) and yellow-blue (+/-b*) color components. The chroma was calculated using Equation 3.4 (Pathare et al., 2013). The chroma (C*) and hue angle (h^*) were calculated using the following Equation 3.4 and 3.5, respectively.

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (3.4)$$

Similar color components have been reported to determine hue angles, which indicated how an eye of human sees the color (Zainuddin et al., 2014). The hue angle was calculated as in Equation 3.5 (Pathare et al., 2013).

$$h^* = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (3.5)$$

Moreover, browning index (BI) indicated the purity of brown color of samples and always used as a reference for browning color changes (Alibaş, 2012). BI was calculated using Equation 3.6 while total color change (ΔE) was calculated using Equation 3.7, where subscript "0" referred to color reading of fresh pomelo peel. Fresh pomelo peel (flavedo, albedo and lamella, pulp waste) were used as the reference and minimal ΔE change denoted minimum color change from the reference material.

$$BI = \frac{[100(x - 0.31)]}{0.17} \quad (3.6)$$

Where $x = \frac{(a^* + 1.7L)}{.(5.645L + a - 3.012b)}$

Red hue signified an angle of 0 ° or 360 ° whilst yellow, green and blue hues showed angles of 90 °, 180 ° and 270 °, respectively. The total color change (ΔE) was determined using Equation 3.7 (Chong et al., 2008).

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (3.7)$$

3.6 Proximate analysis

3.6.1 Determination of moisture content

Moisture content is the quality of water contained in a material. Moisture content was determined using the Standard Official Methods of Analysis of the AOAC

(2000) Method 925.10. This involved drying to a constant weight at 105 °C and calculating moisture as the loss in weight of the dried samples. The aluminum dish was thoroughly washed and dried in an oven at 100 °C for 30 min and allowed to cool inside desiccators. After cooling, they were weighed using a weighing balance and their various weights were recorded as initial weight of empty crucible (W1). Then, 2.0 g of the finely-ground samples were put into the crucibles and weighed to determine initial weight of filled crucible (W2). Thereafter, the sample and crucible were placed inside the oven and dried at 100 °C for 4 h, then cooled and weighed at the same temperature for 30 min until constant weights were obtained to get final weight of filled crucible (W3). Besides, the drying time can be measured by weighed and reweigh the W3 until two or three successive weight is not more than 0.1 - 0.2 mg per 5.0 g of sample. Then, the moisture content of the samples was calculated using Equation 3.8.

$$\frac{(W2)-(W3)}{(W2)-(W1))} \times 100 \qquad (3.8)$$

Where; Initial weight of empty crucible (W1); Initial weight of filled crucible (W2); Final weight of filled crucible (W3)

3.6.2 Determination of ash content

Ash refers to any inorganic materials, such as minerals that present in the fruits. The ash in the fruits can help to change the pH of the urine. It is the inorganic residue which remains after burning process of organic compounds in food in furnace chamber at temperature of 500-700 °C. Total ash content of the samples was determined using furnace incineration, as described by AOAC (2000) Method 900.02, based on the vaporization of water and volatiles with burning organic substances in the presence of oxygen in the air to carbon dioxide at a temperature of 550 °C (dry ashing). About 1.0 g of finely-ground dried sample was placed in a porcelain crucible and incinerated at 525 °C for 6 hr in an ashing muffle furnace (KSL-1700X, MTI Corporation; USA) until ash was obtained. The ash was cooled in a desiccator and weighed. The percentage of ash content in the samples was calculated using Equation 3.9.

$$\frac{Weight\ of\ ash}{Weight\ of\ original} \times 100 = \%\ \text{Ash} \quad (3.9)$$

3.6.2 Determination of crude protein and nitrogen contents.

Protein is a molecule made up of amino acids that are needed for the body to function properly. The protein of fruits can help to reduce the cholesterol and risk of heart disease when regularly eaten. The crude protein content of the samples was determined using the Kjeldahl method (AOAC, 2000, Method 2001.11) which involved protein digestion and distillation. For the protein digestion, about 2.0 g of the sample was weighed into a Kjeldahl flask, and 2 tablets of Kjeldahl Catalyst were added. This was followed by adding 25 mL of concentrated sulfuric acid. The whole mixture was subjected to heat in the fume cupboard. The heating was done gently at first and increased with occasional shaking until the solution turned into green. The temperature of the digester remained above 420 °C for about 30 min. The solution was cooled and black particles found at the neck of the flask were washed down with distilled water. The solution was reheated gently at first until the green color disappeared. Then, it was allowed to cool. To prepare for protein distillation, the Kjeltec distillation apparatus (KjeltecTM 2300, Foss Analytical; Denmark) was steamed through for 15 min, after which a 100 mL conical flask containing 5 mL of boric acid/ indicator was placed under the condenser so that the condenser tip was under the liquid. About 5.0 mL of the digest was pipetted into the body of the apparatus via a small funnel aperture. The digest was washed down with distilled water, followed by the addition of 50 mL of 60 % NaOH solution. The digest in the condenser was steamed through for about 1 to 5 min until enough ammonium sulfate was collected. The receiving flask was removed and the tip of the condenser was washed down into the flask, and then the condensed water was removed. The solution in the receiving flask was treated with 0.01 M hydrochloric acid. Also, a blank was run through along with the sample. After titration, the percentage of nitrogen was calculated using Equation 3.10.

$$\%N = (V_1 - V_2) \times (\text{molarity of acid}) \times 0.01410 \times \text{W} \times 100 \quad (3.10)$$

where; V_1 is the volume of acid used in the titration, V_2 is the corresponding amount of acid for the blank titration, and W is the weight of the sample. On average all biological proteins contain 16 % *N*; therefore protein content is estimated by multiplying *N* % (6.25) is the reciprocal of 0.16). Thus, crude protein does not differentiate between *N* in feed samples coming from true protein or other non-protein nitrogen (NPN) compounds, nor does it differentiate between available and unavailable protein. Then the crude protein was calculated using Equation 3.11.

$$\%N \times 6.25 = \%\ \text{Crude Protein} \tag{3.11}$$

3.6.3 Determination of crude fat content

Fat is the most concentrated source of calories. The total fat in the sample was determined according to the Soxhlet extraction method using Soxtec Extraction (SoxtecTM 2050, Foss Analytical, Denmark). First, a 250 mL clean aluminum cup was dried in an oven at 105 to 110 ℃ for about 30 min and cooled in a desiccator. Approximately 1.0 g of sample was weighed into labeled thimbles. The aluminum cup was weighed correspondingly and filled with about 80 mL of petroleum ether (boiling point 40 to 60 ℃). The extraction thimbles were plugged tightly with cotton wool. The Soxtec apparatus was assembled and allowed to reflux for 75 min. The thimble was removed with care, and petroleum ether was collected from the top container and drained into another container for reused. After that, the flask was dried at 105 to 110 ℃ for 1 hr, when it was almost free of petroleum ether. After drying, it was cooled in a desiccator and weighed (Pearson, 1976). Then, the fat percentage of the samples was computed using Equation 3.12 (AOAC, 2000; Method 960.39).

$$\frac{\text{Weight of fat}}{\text{Weight of sample}} \times 100 = \%\ \text{fat} \tag{3.12}$$

3.7 Antioxidant capacity

3.7.1 Extraction of sample

The extraction process is a process where a solvent used to extract the phenolic compounds from the samples (M'hiri et al., 2014). The samples were extracted based on a method described by Toh et al. (2013). Briefly, 0.5 g of ground sample was weighed and added with 10 mL of 80% methanol (v/v). The mixture was placed in a conical flask wrapped with an aluminum foil and shaken at 200 rpm at 30 °C for 3 hr on an orbital shaker (Unimax 1010, Heidolph, Germany). The extract was then filtered through a filter paper (Whatman No. 1) to obtain a clear solution. This step was repeated twice. The filtrate was used for determination of total phenolic and antioxidant capacity.

3.7.2 Determination of total phenolic content (TPC).

This assay is regularly applied and measured the phenolic antioxidants or phenolic content that present in the dried fruits peels. Secondary metabolite also known as phenolic compounds from plants are significant to scavenge a free radical and possesses multiple medicinal and physiological functions (Manach, et al., 2005; Sultana, et al., 2012). Total phenolic content (TPC) of the sample was determined by Folin-Ciocalteau assay, which was based on a method of (Singleton & Rossi, 1965) with modification by Ikram et al. (2009). Briefly, an aliquot (0.2 mL) of diluted extract was mixed with 1.5 mL of 10-fold diluted Folin-Ciocalteau reagent. The mixture was allowed to stand at room temperature for 5 min. Then, 1.5 mL of 6 % sodium carbonate solution was added to the mixture. The mixture was homogenized and allowed to stand at room temperature for 90 min. The absorbance of the reacting mixture was measured at 725 nm against a blank using a spectrophotometer. All samples were analyzed in triplicate. A standard curve for quantification was plotted using gallic acid (0 - 0.2 mg/mL) and the results were expressed as mg gallic acid equivalent (GAE) per 100 g fresh weight (FW).

3.7.3 DPPH

Promising antioxidant activity of the peel's extract can be evaluated by the reduction of DPPH radical (Huang et al., 2005). Evaluation of free radical scavenging activities of material has been widely explored by using a model of scavenging the stable DPPH radicals (He et al., 2012). DPPH Assay Free radical scavenging activity of a sample was determined using 2,2- diphenyl- 1- picrylhydrazyl (DPPH) method (Jongaroontaprangsee et al., 2014;Turkmen et al., 2005). An aliquot of 1.5 mL of 0.2 mM DPPH radical in methanol was added to a test tube with 0.5 mL of the sample extract. The reaction mixture was vortex-mixed for 30 s and left at room temperature in the dark for 30 min. The absorbance was measured at 517 nm using a UV-VIS spectrophotometer. The total antioxidant activity was expressed as the percentage of inhibition of DPPH radical and was determined by the following Equation 3.13.

$$\%\ Antioxidant\ activity = \frac{Abs_{control} - Abs_{sample}}{Abs_{control}} \times 100 \qquad (3.13)$$

Where; $Abs_{control}$ is the value of absorbance without sample known as control, Abs_{sample} is the value of absorbance with sample

3.7.4 Ferric reducing antioxidant power (FRAP) assay.

This assay is considered as a one of the useful indicators to measure antioxidant activity of the peel's extract (Ismail et al., 2013) and was performed according to a method described by Benzie and Strain (1996). The FRAP reagent was consisted of 300 mM acetate buffer (pH 3.6), 10 mM 2, 4, 6-Tri (2-pyridyl)-s-triazine (TPTZ) in 40 mM HCl, and 20 mM Ferric Chloride Hexhydrate (FeCl3·6H2O). The FRAP reagent was prepared by mixing the acetate buffer, TPTZ solution, and FeCl3·6H2O solution in proportion of 10:1:1 (v/v/v). Briefly, an aliquot of appropriately diluted sample (1 mL) was mixed with 3 mL of freshly prepared FRAP reagent and mixed thoroughly. The reaction mixture was incubated at 37 °C for 30 min. Absorbance of the mixture was measured at 593 nm versus blank. Ferrous sulphate (0 - 1000 mM) was used to plot a

calibration curve for quantification, and the results were expressed as mmol Fe^{2+} per 100 g FW. All samples were analyzed in triplicate.

Different parts of pomelo residues (Flavedo, albedo, lamella and pulp waste) were undergone physicochemical and antioxidant properties (section 3.5 till section 3.7). Nevertheless, in order to select the best parts of pomelo residues, current study only focused on the highest value of total phenolic content (Section 3.7.2) as it is considered the main finding of the current study. Then, the selected part was undergo the best drying selection using response surface methodology using factorial design application in section 3.8.

3.8 Selection of the ideal drying process of selected pomelo residues

A selected two factor, 3 level factorial design was used for the experiment in first stage. Freeze drying is not to be considered in the selection of drying methods due to the operation conducted at single temperature and involve lyophilization process whereby the water is frozen prior to drying that followed by sublimation and desorption process (Nireesha et al., 2013). Thus, conventional hot air drying, and vacuum oven drying applies straight forward drying from fresh form at different drying temperatures (50,60, 70, 80 and 90 °C) without frozen. The current objective to determine the effect of different drying temperature on the desired quality (MC, TPC and DPPH) were conducted. The selection of drying temperature has been explained in Section 3.3.1. The effect of two independent variables, drying methods (CD and VD) and drying temperature (50-90 °C), on three selected response variables, moisture content, TPC and DPPH was evaluated (Table 25).

Table 25 Factor levels of the full factorial design used in the RSM study of the drying conditions

Independent variables	Symbols		Levels	
	Natural	Coded	Natural	Coded
Drying modes	D	X_1	Conventional drying (CD)	0
			Vacuum drying (VD)	1
Temperature	T	X_2	50 ℃	-2
			60 ℃	-1
			70 ℃	0
			80 ℃	1
			90 ℃	2

The best version of drying process was performed using multivariate response method (Abano et al., 2015) with Equation 3.14

$$DI = \left[\prod_{i=1}^{3} di(Y_i)\right]^{\frac{1}{3}} \quad (3.14)$$

Desirability index (DI) represents the desirability for the various response: Moisture content, total phenolic content (TPC), and DPPH (Y_i). The DI ranges between 0 and 1. Zero is the least preferred value while 1 is the most desired. Maximizing DI is the goal of optimization analysis. The optimization process includes goals and priorities for the factors and the responses. For this current study, the goal for the factors was at any level within the range of the design values, however, in the case of the responses, minimum values of moisture content (MC) (less than 10%) , maximum total phenolic content (TPC) and percentage inhibition of DPPH (%) were desired.

Pomelo albedo obtained from section 3.7.2 were further on undergo analysis for mathematical modelling in section 3.9 and the best drying condition of the selected parts were used to undergone kinetic degradation during storage in section 3.11.

3.9 Mathematical modelling.

3.9.1 Drying Kinetics and Data Analysis.

The drying kinetic curve and drying rate curve were presented as the experimental results from this study. During drying treatment, for every 10 min interval, the sample (2.0 g) was taken out and rapidly weighed on a digital balance with the accuracy of ± 0.0001 g (Model XP204, Mettler Toledo, USA). Then, the samples were taken back to the oven directly. The action was completed in approximately 30 s. The weight were recorded accordingly until two or three successive weight is not more than 0.001 g of sample. Drying kinetic was created based on the variation of moisture ratio (MR) as a function of time (t) (Tasirin et al., 2014). Moisture ratio (MR) was used as a dependent variable as described in Equation 3.15, so that the drying curves for all experiments were comparable.

$$MR = \frac{M_t - M_e}{M_i - M_e} \tag{3.15}$$

The dependent variables were initial moisture content (M_i), equilibrium moisture content (M_e) and moisture content in actual time (M_t). Nonetheless, in this study, the equilibrium moisture content was relatively small (less than 0.001 g) compared to M_i or M_t, and nearly approached the dry matter content, therefore, it was assumed negligible. This approximation was also used by Tasirin et al. (2014) and (Onwude et al., 2016) where they simplified the Equation 3.15 to Equation 3.16.

$$MR = \frac{M_t}{M_i} \tag{3.16}$$

The fraction of moisture content of the pomelo residues (dry basis) was calculated using the previously described in Equation 3.16. Torki-Harchegani et al. (2016) has suggested using semi-theoretical models to describe the drying kinetics of agricultural materials. In this work, the experimental data obtained were fitted to seven models (Table 26).

Table 26 Mathematical models applied to the drying curves**.**

Model no	Model name	Model expression
1	Henderson and Pabis	$MR = a\exp(-kt)$
2	Logarithmic	$MR = a\exp(-kt) + b$
3	Wang and Singh	$MR = 1 + at + bt_2$
4	Newton	$MR = \exp(-kt)$
5	Midilli and Kucuk	$MR = a\exp(-kt^n) + bt$
6	Two-term exponential	$MR = a\exp(-k_1t) + b\exp(-k_2t)$
7	Diffusion approach	$MR = a\exp(-kt) + (1-a)\exp(-kbt)$

The parameters a, c, k, and n were calculated by non-linear regression analysis.
Source: Torki-Harchegani et al. (2016)

3.9.2 Fitting Models to Experimental Data.

The seven models listed in Table 26 were fitted to the experimental data. Non-linear regression analysis was performed using Scientific Data Analysis and Graphing Software (SigmaPlot) (Systat Software Inc., Chicago, IL, USA). The current study only focuses on fitting the existing drying mathematical models. The goodness of fit between the predicted and experimental data was evaluated based on the coefficient of determination (R^2), standard error of estimation (SEE), root mean square error (RMSE). The higher the coefficient of determination (R^2) and the lower the SEE and RMSE, indicating a good fit (Torki Harchegani et al., 2016) to the data. The R^2, SEE and RMSE were calculated using Equation 3.17, 3.18 and 3.19 respectively:

$$R^2 = 1 - \left[\frac{\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2}{\sum_{i=1}^{N}\left(MR_{pre} - MR_{exp,i}\right)^2}\right] \quad (3.17)$$

$$SEE = \sum_{i=1}^{N}\frac{\left(MR_{exp,i} - MR_{pre,i}\right)^2}{d_f} \quad (3.18)$$

$$RMSE = \sqrt{\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2} \quad (3.19)$$

where $MR_{exp,i}$ and $MR_{pre,i}$ are the experimental and predicted moisture ratio, respectively; N is the number of observations; and z is the number of constants in the models. The equation with the highest R^2, low x^2 and RMSE values was chosen as the best mathematical model to describe the drying curves of pomelo residues.

3.9.3 Moisture effective diffusivity

Effective diffusivity can be determined by using the drying data based on sufficiently long drying times of the Fick's equation (Crank, 1975; Torki-Harchegani et al., 2016). Equation 3.20 assumes that effective diffusivity (D_{eff}) is constant and the shrinkage of the sample is negligible.

Equation 3.20 is obtained when one-dimensional mass transfer was assumed as major mechanism occurred in the slab geometry. Also the conditions were assumed:

- At the beginning of drying, moisture was uniformly distributed inside the peels
- Constant of diffusion coefficient
- Shrinkage was not significant as outer resistance to moisture transfer (Crank, 1975; Doymaz, 2015; Tuncer et al., 2020).

When long drying times are concerned, the first term can be taken into consideration by itself and the equation is formed as Equation 3.21.

$$MR = \frac{M_t - M_e}{M_i - M_e} = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n+1)^2}\ exp\left(\frac{-(2n+1)^2\pi^2 D_{eff}t}{4l^2}\right) \quad (3.20)$$

It can be further simplified to a straight line (Equation 3.21):

$$MR = \frac{8}{\pi^2}exp\left(\frac{-\pi^2 D_{eff}t}{4l^2}\right) \quad (3.21)$$

In the equation l expresses the half thickness of peel assumed as slab(m). So as to determine the D_{eff} value, the drying time dependent change of the ln (MR) values (Equation 3.22) as are plotted graphically and D_{eff} value achieved via the straight-line slope

$$\ln(MR) = \ln(\frac{8}{\pi^2}) - D_{eff}\left(\frac{\pi}{2l}\right)^2 t \quad (3.22)$$

The value of the slope (K_s) can lead to an identification of D_{eff} at different temperatures as in Equation 3.23:

$$K_s = -D_{eff}\left(\frac{\pi^2}{4l^2}\right) \quad (3.23)$$

The effective diffusivity varies with the temperature according to Arrhenius dependence as in Equation 3.24:

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{RT}\right) \quad (3.24)$$

where D_0 is diffusivity at an infinite temperature (m^2/s), E_a is the activation energy for moisture diffusion (kJ/mol), T is the drying temperature (Kelvin) and R is the gas constant (8.314 J/mol K) as in Equation 3.25.

$$\ln D_{eff} = \ln (D_0) + \left(-\frac{E_a}{R}\right)\frac{1}{T} \quad (3.25)$$

3. 10 Determination phenolic compounds using high performance liquid chromatography (HPLC).

Samples were filtered through a 0.45m pore size membrane before injection. An Agilent 1100 HPLC system (Agilent Technologies, Palo Alto, CA, USA) operated by Windows NT based Chem Station software was used. The HPLC equipment was used with a diode array detector (DAD). The system consisted of a binary pump, degasser and auto sampler. The column used was a Poroshell 120 C18 column (Waltham, MA, USA): 4.6 x 100mm x 4µm. The injection volume of the extract was 10µL per sample. The mobile phase consisted of two solvents: Solvent A, 1% Trifloroacetic acid (TFA) in 90% of 50 mM Phosphate buffer, and Solvent B, 10% Acetonitrile (ACN). Phenolic compounds were eluted under the following conditions (Shah et al., 2013; Sintuya et al., 2018) with modifications: gradient conditions 0 to 10% solvent B (0 mins), 10 to 40% solvent B (2 to 4 mins), 40 to 10% solvent B (4 to 7 mins), followed by washing and reconditioning of the column. The separations were performed with a flow rate of 1mL/min, which was directly injected in the ESI source, without any splitting. The column temperature was maintained at 25°C. The analysis time was of 7 minutes. The HPLC method was tested on specific major phenolic compound (naringin) present in samples. The polyphenols standard solutions (5- 200 µg/mL) were prepared in methanol. The ultra-violet-visible-spectra (scanning from 200 to 300nm) were recorded for all peaks. Triplicate analyses were performed for each sample. The identification of phenolic compounds was obtained by using authentic standards while quantification was performed based on standard curve at different concentration.

3.11 Effects of storage on phenolic compounds and antioxidant capacity.

Storage and drying processing highly influenced the phenolic composition and their antioxidant capacity (Gil-Izquierdo et al., 2001). It was highly suggested to evaluate the changes of storage time in order to check the shelf life of the desired processing at selected drying temperature and compare directly among the part of pomelo residues. Previous study by Sirisomboon & Lapchareonsuk (2012) on fresh pomelo fruits stored in a cooled room (10 °C) clearly reported that the pomelo fruits should not be stored for more than 75 days.

The present study only investigated the dried extract of pomelo albedo containing bioactive compound with antioxidant properties. For the storage test, the dried crude extract in petri dish was stored in the dark (dark box) immediately in temperature-controlled storage chiller at 8°C for 3 months.

Prior to each analysis, the crude extract (0.2g) were equally divided into small plastic tube (1.5 mL). It was covered by aluminum foil to avoid contact with UV-light and separated to different analysis. Samples analysis were carried out for TPC, DPPH and FRAP at different interval time. Each analysis was undergone in laboratory with the lamp covered by red plastic to avoid the UV lamp effects the results. Total phenolic content was quantified via Section 0. Major phenolic compound (naringin) of the dried extracts was quantified using HPLC (Section 0).The corresponding antioxidant properties of the dried extracts was determined with DPPH radical scavenging assay and FRAP assay were carried out. All subsequent analyses were executed every 7 days for a storage period of three months.

3.11.1 Degradation Kinetic Studies

In general, variation in quality factor "C" under isothermal conditions can be described by Wang et al. (2004) in Equation (3.26).

$$\frac{dC}{dt} = -k(C)^n \tag{3.26}$$

Where *k* is the rate constant, *C* is the quantitative indicator of a quality attribute at time, *t* and *n* is the order of reaction.

The degradation kinetics of total phenolic content and antioxidant activity was evaluated using zero-order, first-order, and second-order kinetic models in Equation 3.27, 3.28 and 3.29 as described by Sintuya et al. (2018):

$$C_t = C_0 - k_1 t \quad (3.27)$$

$$C_t = C_0 \times exp^{-k_1 t} \quad (3.28)$$

$$\frac{1}{C_t} = \frac{1}{C_0} - k_1 t \quad (3.29)$$

Where k_1 is the degradation rate constant, C_0 is the antioxidant activity when $t = 0$, and C_t is the antioxidant activity at any given time.

Half-life ($t_{½}$) is the estimated time required for the antioxidant activity of the phenolic compounds to decrease by 50% from its initial value ($C = 0.5C_0$). Half-life of antioxidant activity at its corresponding drying method is determined using the equation 3.30. It was determined using equation 3.30 (zero order) or equation 3.31 (first order) or equation 3.32 (second order) depending on the best fitted to the experimental data:

$$t_{\frac{1}{2}} = \frac{[C]_0}{2k_0} \quad (3.30)$$

$$t_{\frac{1}{2}} = \frac{\ln 2}{k_1} \quad (3.31)$$

$$t_{\frac{1}{2}} = -\frac{1}{k_2[C]_0} \quad (3.32)$$

Where $t_{(1/2)}$ is the half-life of reaction; C_0 is the antioxidant activity at time 0; k_0, k_1, k_2, is the antioxidant degradation rate constant

3.12 Statistical analysis

The data obtained from the experiments were then subjected to statistical analysis using IBM SPSS Statistics 21.0 edition where One-way ANOVA uses Duncan's

test was performed to evaluate the significant difference between mean values. The significant difference was considered at confidence limits of 95 % ($p < 0.05$). Each analysis was performed in triplicate, and the data were represented as the mean of three independent experiments. All mathematical data analysis and kinetic modelling was conducted using nonlinear regression performed by statistical methods via Scientific Data Analysis and Graphing Software (SIGMA PLOT, 12.0). Further details on summary of analysis involved and type of sample being used were summarized in Appendix C (Table C1). Paired t-test was used to assess the differences between the paired groups while $p > 0.05$ signifies no significant differences. Post hoc multiple assessments were made by least significant difference (LSD), comparisons have made at 5% of the level of significance. Pearson correlation coefficient was determined between the content of the antioxidant compound and dried pomelo residues. The p-value of less than 0.05 was considered significant differences between different pomelo samples. The different column of Duncan tables was signifying as significantly different between drying methods which can be referred to Appendix D.

CHAPTER 4
RESULTS AND DISCUSSIONS

4.1 Composition of different parts of pomelo residues

Table 27 presents the composition of each part of pomelo fruits. The samples were taken from the Department of Agriculture in Kinta district, Perak, Malaysia. The weight of the Tambun White (PO52) pomelo ranges from 1500 g to 2500 gram but researchers have reported weights ranging from 897.5 g - 2400g for different pomelo varieties in Thailand (Pichaiyongvongdee & Haruenkit, 2009). The weight of pomelo fruit can vary even for the same cultivar, due to dissimilarities in nutrition and growing environment (Rahman et al., 2003; Pichaiyongvongdee & Haruenkit, 2009).

Table 27 Compositions of parts of pomelo residues

Yield / Parts of pomelo fruits	% per whole fruit (g/100g)	% per whole pulp (g/100mL)	% per total residues (g/100g)
Peels			
#Flavedo	8.22 ± 0.69^{c}	nd	15.56 ± 0.64^{c}
#Albedo	20.20 ± 4.95^{b}	nd	37.76 ± 6.51^{a}
#Lamella	8.05 ± 0.65^{c}	nd	15.39 ± 2.38^{b}
Pulp	62.60 ± 5.21^{a}	100	nd
Juice (w/v)	nd	64.93 ± 7.15^{a}	nd
#Pulp waste(w/w)-after juice extraction	16.39 ± 0.65^{b}	26.29 ± 1.73^{b}	31.28 ± 3.99^{a}
Total of wastes except pulp	52.86		

Note: nd = not determine; Values are mean (n = 5) ± SD, significant different ($p < 0.05$) of data were represent with the letter (a,b,c) within the column

this symbol represents pomelo wastes

The cross section of each part of fruits from the flavedo, albedo, lamella and pulp waste (Figure 2) were analyzed for composition of different parts of pomelo residues. This study focuses on pomelo residues that include pomace or residues obtained after the juice has been extracted and readings can reach approximately 52.86 g/100g of whole fruit which contains up to 50% of waste. Thus, the yields of each part of waste are significant ($p < 0.05$) to be considered to fully utilize the waste produced by pomelo fruit juice industries. The highest weight is in the albedo (20.20 g/ 100g whole fruit) and the percentage of albedo is 38 g/100g from the total of pomelo residues. The second highest net weight is from pulp waste after juice extraction (16.39g/100g pulp) similar to 31.28 g/ 100g of total residues obtained from remaining residues after the juice have been extracted. The net weight from flavedo (8.22g/100g whole fruit) is equivalent to 15.56g/100g of total residues and the net weight from lamella (8.05g/100g whole fruit) is similar to 15.39 g/100g of total residues which is not significant ($p > 0.05$). Knowledge on the exact yields from different parts of pomelo residues provide valuable information about the production of waste yields per year from pomelo fruit production (Section 2.1).

4.2 Effects of drying methods and temperature on physiochemical properties of pomelo residues

4.2.1 Effects of drying methods and temperature on moisture content

Table 28 shows the effects on MC during post-drying using treatments FD, CD and VD on pomelo residues. Freeze dried pomelo residues were used as a control group. Reduction of MC (less than 10%) is necessary to extend the shelf life of the product as well as to prevent enzyme and microbial activities (Abirami et al., 2012).

Table 28 Effects of drying methods on moisture content of pomelo residues at different drying temperature

Drying method	Temp. (°C)	Flavedo	Albedo	Lamella	Pulp waste
Fresh		80.59 ± 0.47^{a}	77.69 ± 1.31^{a}	78.18 ± 0.51^{a}	82.90 ± 0.47^{a}
FD		9.33 ± 0.26^{d}	8.63 ± 0.31^{d}	5.67 ± 0.22^{h}	9.63 ± 0.57^{d}
CD	50	10.18 ± 0.59^{c}	6.53 ± 0.55^{f}	5.85 ± 0.37^{gh}	10.07 ± 0.63^{d}
	60	5.15 ± 0.48^{fg}	5.88 ± 0.27^{f}	6.33 ± 0.20^{fg}	3.48 ± 0.30^{ij}
	70	6.81 ± 0.86^{e}	3.33 ± 0.48^{h}	4.55 ± 0.34^{ij}	2.90 ± 0.71^{j}
	80	4.05 ± 0.12^{h}	3.72 ± 0.31^{h}	4.79 ± 0.06^{i}	5.74 ± 0.19^{h}
	90	4.85 ± 0.47^{g}	4.64 ± 0.39^{g}	4.07 ± 0.08^{j}	3.89 ± 0.59^{i}
VD	50	16.60 ± 0.33^{b}	20.26 ±0.31^{b}	19.44 ±0.48^{b}	22.31 ± 0.23^{b}
	60	10.80 ± 0.06^{c}	9.72 ± 0.13^{c}	15.34 ±0.69^{c}	14.93 ± 0.09^{c}
	70	8.66 ± 0.22^{d}	8.95 ± 0.19^{cd}	7.82 ±0.031^{d}	8.67 ± 0.24^{e}
	80	6.51 ± 0.10^{e}	7.58 ± 0.15^{e}	6.80 ±0.33^{ef}	7.31 ± 0.26^{f}
	90	5.71 ± 0.18^{f}	8.57 ± 0.24^{d}	7.32 ±0.14^{de}	6.46 ± 0.07^{g}

Moisture content was determined based on g moisture/100g dry weight (%)

Values are mean ± SD, significant different ($p < 0.05$) of data were represent with the letter (a,b,c) within the column

FD: Freeze drying; CD: conventional drying; VD: Vacuum drying.

For FD the moisture content of the flavedo significantly reduced ($p < 0.05$) from the fresh form (80.6%) to 9.3%, and for CD it is within the range of 5.15% - 4.05%, while for VD it is within the range of 10.8% - 5.71% at different drying temperatures (50 °C - 90 °C). There was a significant reduction ($p < 0.05$) of MC after CD (5.15 - 4.05%) at 60 – 90 °C and VD (6.51 – 5.71%) at 80 - 90 °C in comparison with FD (9.33%). It might be due to the mode of moisture loss between two different drying methods which affect the final MC of flavedo. FD encompasses water evaporation by sublimation and the conversion of water from solids to vapor phase in a vacuum condition (Dzung, 2012). Meanwhile, in conventional drying the different drying

temperatures (50 °C – 90 °C) applied corresponds to simultaneous water evaporated via heat and mass transfer (Vega-Gálvez et al., 2009). FD produced significantly greater MC ($p < 0.05$) than CD for flavedo (Refer to Appendix D, Table D2) which might be due to the ice crystals formed (Singh & Heldman, 2001) throughout freezing phase that were not completely removed during freeze drying. The results of CD method of the current study (6.81 - 4.05%) are comparable with previous researches on peels of *C. maxima* (8.05 - 8.66 %) (Abirami et al., 2014; Mat Zain et al., 2014).

The VD of flavedo (16.6 – 10.8 g/100g DW) showed higher MC particularly at lower drying temperature (50 °C and 60 °C correspondingly) compared to control (FD). During vacuum drying process, flavedo was dried in reduced pressure environment, leading to an oxygen-deficient condition (Wang et al., 2018). Indirectly, the boiling point (vaporization temperature) of material which required the moisture to be removed was reduced (Parikh, 2015) as well. In the present study, condensed moisture was discovered on vacuum dryer's glass wall during drying which required longer time of drying. However, as the current study focused on the 24 hr of drying time, the MC of the flavedo recorded higher retention of MC with more than 10% differences at lower temperature (50 °C and 60 °C), as shown in Table 28. Furthermore, the heat transfer in vacuum drying in conduction method, the heat was transferred from the heater plate to the product, compared to conventional drying which applied mode of heat transfer based on convection. This method is similar with previous studies done by Xu et al. (2017). Therefore, the VD flavedo results in higher MC retained in comparison with CD flavedo.

Secondly, for the albedo, the whitish color of fresh albedo contained higher MC (77.7%) which has been reduced to 8.6% during FD, within the range from 6.8% – 3.3% during CD and within the range for 20.3% - 7.6% during VD at different temperatures (50 ° C – 90 °C). Post-drying treatment significantly impacts ($p < 0.05$) the MC of albedo (Refer to Appendix D, Table D3). In brief, the CD (6.8% – 3.3%) albedo showed significantly lower ($p < 0.05$) MC value compared to FD (8.6%) and VD (20.3% - 7.6%) albedo, which is lesser than pomelo albedo reported by Mat Zain et al. (2014) (16.13%) and for orange peel (9.46%)(Nassar et al., 2008). Therefore, conventional

drying results in rapid moisture removal from air filled intercellular space of albedo (Thielen et al., 2015), thus decreasing the MC.

As temperature increases, the trend of MC tends to decrease in both drying methods (CD and VD) for albedo. As suggested by Geankoplis (2003), the MC containing lower value than 10% is recommended and considered a safe MC level to prevent undesired changes caused by microorganisms that can result in spoilage. Lesser MC can be advantageous for shelf life (refer to Section 4.5) to retain the quality of the product. This result is in contrast with VD at lower temperature (50 °C), the MC of VD (20.26%) was observed to be higher than the recommended level (10%).

Studies have shown that conventional drying and vacuum drying applied different operation despite both using heater as medium to remove the moisture. The fact that heat was transferred to the product in limited amounts in vacuum drying compared to conventional drying could be the reason that the MC was still found to be higher in VD albedo. The contact surface between the hot air stream (CD) with the product by convection and the heating plate (VD) by conduction significantly affects ($p < 0.05$) the rate of moisture removal during drying. This is because of higher surface area (Appendix E; Figure E1 (A)) of CD albedo was exposed to the hot air stream, while VD albedo was exposed for a limited time (refer section Appendix E; Figure E1 (B)) to the heated tray by conduction. Lower rate of evaporation process might occur during vacuum drying process. Thus, this could affect the rate of moisture removal resulting in higher MC value in VD albedo. Similar trend was discovered based on previous study done by Wang et al. (2018) and Xu et al. (2017).

Thirdly, for the lamella which cover the juice sacs, results of MC by CD were within the range from 4.1% to 6.3% and by VD results were within the range from 19.4% to 6.8%. Post-drying (CD) of lamella (4.6 – 4.1%) showed a significant reduction ($p < 0.05$) of MC compared with FD (5.7%) at higher drying temperature (70 – 90 °C). Nevertheless, FD lamella (5.7%) showed significantly lower ($p < 0.05$) than CD lamella (5.9 – 6.3%) dried at lower temperature (50 – 60 °C). It might be due to the application of vacuum in the freeze drying process which leads to high vapor pressure differential between the surface and the centre of the product and allowing higher rate of moisture removal in comparison to CD (Geankoplis, 2003). In contrast, the findings of

VD lamella (19.4% and 15.3%) at lower drying temperature (50 °C and 60 °C) showed similar trend with the albedo and flavedo, that is more than 10%. This indicates that the moisture on the surface of the lamella evaporates easily and quickly, but the bound moisture inside the samples could not travel to the surface simultaneously due to the crust and film formation on the surface samples which eventually limit the evaporation of moisture (Xu et al., 2017). In addition, dried lamella exhibited lower MC than the peel fibre (7.1%, 7.3%), peel (8.1%,8.2%) and pulp (8.5%,7.8%) of different variety of *Citrus maxima* (red) and *Citrus maxima* (white) respectively, which were dried overnight (24hr) at 65 °C (Abirami et al., 2014).

Lastly for pulp waste, the remaining pomace during post extraction exhibited MC for FD at a value of 13.95%, for CD at a value of 10.1 – 2.9% and for VD at a value of 22.3 - 6.5% at different drying temperature (50 °C – 90 °C). Clearly, FD pulp waste results in higher MC than the recommended level of MC (Table 4.2). The samples in the current study were frozen prior to freeze drying which lead to formation of ice crystals made upon the frozen phase. These ice crystals were not completely thawed, and this leads to higher levels of MC (Singh and Heldman, 2001). Then, the sublimation (solid changed to vapor phase) process happened under vacuum condition (Dzung, 2012). The CD pulp waste (5.74 - 2.90 %) showed significant reduction ($p < 0.05$) of MC compared to VD (22.31-6.46%) and FD (9.63%), which conformed to the recommended levels (10%). The reduction of the MC is likely to be initiated by exposure to oxygen and heated air resulting in higher drying rate and evaporation of moisture in CD compared to VD (Nunes et al., 2016).

As can be seen, the MC of VD pulp waste within range of 22.31 to 6.46% gradually declined as temperature is increased (50 °C to 90 °C). Reduction of MC for pulp waste due to the vapor pressure gradient between the product and the environment (partial vacuum in the drying chamber) particularly on the product surface (Dev & Raghavan, 2012; Karam et al., 2016). In addition, MC less than 10% is recommended as microbial was inactive at lower MC to avoid the spoilage from occurring. Furthermore, the trend of the results are comparable with the results for dried lemon peels (4.31%) which showed that MC is affected by different drying methods (Nesrine et al., 2015). Thus, CD at different drying temperature (60 °C – 90 °C)

show MC (5.74 - 2.90%) and VD at 70 ℃ - 90 ℃ (8.67 – 6.46%) significantly lower (p < 0.05) the MC than the control (FD) (9.63%).

The MC of CD pulp waste at 80 ℃ higher (5.74%) than at 70℃ (2.90%) due to higher oxidation of unsaturated fatty acids (fat content). This is consistent with the value of fat content at 80 ℃ is lower (0.77%) than at 70 ℃ (1.17%) in Table 29) generated carbon dioxide and water (Bradley, 2010). This indicates lower the fat content, higher the MC (Table 28). Higher value of MC (5.65 %) also were observed in a study done by (Issis et al., 2019) when high drying temperature (80 ℃) applied compared to lower drying temperature (70 ℃) resulted was 5.64% using vacuum drying on maqui berry. Moreover, drying kinetics of 80 and 90 ℃ shows no increment of moisture ratio (refer Figure 13), which reflects the phenomena of case-hardening effect. This effect prevents the water release and slows down the drying rate and drying performed at higher temperature does not improve the drying rate (Garau et al., 2007).

Overall, moisture content is a significant parameter for dried products, as it can be used to determine whether the final product is stable and safe from microbial and enzymatic reaction. Less than 10% MC is a safe level of MC for dried product (Geankoplis, 2003). As expected when the drying temperature increases, the MC tends to decrease significantly (p < 0.05) in all different parts of pomelo peels. Significantly lower moisture content in CD than VD was discovered at different drying temperatures. In terms of different drying operation, the lowest to highest value of MC was observed in CD followed by FD and VD in overall parts of pomelo residues. Under FD parts of pomelo residues in order of lowest to highest value retained after drying was lamella< albedo<flavedo< pulp waste.

4.2.2 Effects of drying methods and temperature on color

Color, a significant parameter for quality indicator is commonly reported for dried products since it influences the acceptability of the product to the consumer. Consumer prefers the color to be fresh (Refer to Appendix F, Table F1) and brighter with minimal change of color (brown) (Barret et al., 2010). The findings on the quality (color and nutritional composition properties) affected by different drying methods at

different drying temperature of flavedo, albedo, lamella and pulp waste are tabulated in Table 29.

4.2.2.1 Flavedo

Post drying significantly ($p < 0.05$) affects the value of L*, a*, h*, C*, BI, and ΔE (Refer to Appendix D, Table D4. Freeze drying was used as control in the current study as it reflects equivalent properties with fresh form of flavedo (Appendix B; Table B1). The L* of FD flavedo (59.6) displayed the highest value compared to CD exhibited L* value within the range of 43.7 to 58.9 and VD from 37.7 to 39.74 respectively. Significant variations ($p < 0.05$) of L* were discovered as temperature increased particularly during conventional drying (CD). In contrast with vacuum drying, L* value of flavedo exhibited no significant difference ($p > 0.05$) at different drying temperature of flavedo. Nevertheless, the value of VD showed significant reduction ($p < 0.05$) of L* value more than 30% generally at 50 - 90 °C in comparison with CD which only reduced by less than 10%. This might be indicated by the development of brown pigments from Maillard reaction and non-enzymatic mechanism or caramelization of the sugar during drying (Mireles-arriaga et al., 2016; Rafiq et al., 2019).

The significant parameters (a) reflect either greenness (negative value) or redness (positive value) of the product. FD flavedo shows negative value (-3.3) which means that the value is similar with fresh value (-7.9) for both drying methods (which in positive value). The value by CD is in the range of 2.8 to 14.0 while by VD the value is from 4.4 to 5.5 which is higher than the control value

Table 29 Quality of pomelo residues affected by different drying methods at different drying temperature

Samples	Drying condition/ Temp.	L*	a*	b*	ΔE	Ash content (%)	Protein content (%)	Fat content (%)
Flavedo	FD	59.63 ± 0.06^{a}	-3.3 ± 0.30^{f}	36.87 ± 0.12^{b}	9.57 ± 1.44^{f}	5.92 ± 0.03^{d}	7.63± 0.06^{h}	1.63 ± 0.05^{a}
	CD50	58.93 ± 0.91^{ab}	2.8 ± 0.07^{e}	37.03 ± 0.76^{b}	13.95 ± 0.67^{ef}	6.45 ± 0.21^{b}	11.64 ± 0.01^{a}	1.27 ± 0.25^{a}
	CD60	54.33 ± 1.14^{d}	4.0 ± 0.21^{d}	34.63 ± 0.12^{c}	14.48 ± 0.26^{de}	6.75 ± 0.21^{abc}	10.82±0.05^{c}	0.73 ± 0.12^{cd}
	CD70	55.77 ± 1.10^{c}	5.6 ± 0.14^{b}	38.53 ± 0.68^{a}	19.37 ± 0.92^{cd}	7.15 ± 0.07^{a}	10.67± 0.11^{d}	0.80 ± 0.10^{c}
	CD80	57.73 ± 1.00^{b}	5.7 ± 0.40^{b}	39.33 ± 0.60^{a}	15.37 ± 1.07^{de}	7.00 ± 0.14^{ab}	11.64±0.12^{a}	0.57 ± 0.12^{d}
	CD90	43.70 ± 1.28^{e}	14.0 ± 0.23^{a}	34.50 ± 0.87^{c}	23.59 ± 1.18^{bc}	6.50 ± 0.71^{bc}	11.31± 0.01^{ba}	0.80 ± 0.10^{c}
	VD50	38.70 ± 0.55^{gh}	4.50 ± 0.10^{c}	27.0 ± 0.40^{e}	31.56 ± 3.81^{a}	5.88 ± 0.17^{e}	8.70 ± 0.02^{g}	0.58 ± 0.02^{d}
	VD60	37.70 ± 0.06^{h}	4.53 ± 0.06^{c}	27.27 ± 0.06^{e}	32.35 ± 3.49^{a}	6.52 ± 0.01^{bc}	9.18± 0.05^{e}	0.68 ± 0.02^{cd}
	VD70	41.74 ± 0.25^{f}	4.43 ± 0.21^{c}	29.43 ± 0.31^{d}	28.21 ± 3.60^{ab}	6.44 ± 0.02^{bcd}	8.89±0.06^{f}	0.85 ± 0.06^{c}
	VD80	39.74 ± 0.21^{g}	4.57 ± 0.06^{c}	29.50 ± 0.26^{d}	29.98 ± 3.64^{a}	6.43 ± 0.08^{bcd}	8.56±0.05^{g}	0.78 ± 0.01^{c}
	VD90	38.44 ± 0.31^{gh}	5.47 ± 0.12^{b}	29.50 ± 0.26^{d}	31.50 ± 3.21^{a}	6.29 ± 0.10^{cd}	8.83±0.01^{f}	1.27 ± 0.02^{b}

Table 29 (Continued)

Samples	Drying condition	L*	a*	b*	ΔE	Ash content (%)	Protein content (%)	Fat content (%)
Albedo	FD	70.13 ± 0.42^{b}	3.37 ± 0.12^{f}	24.63 ± 0.42^{h}	7.64 ± 0.16^{h}	2.64 ± 0.55^{c}	3.49±0.08^{de}	0.60 ± 0.05^{c}
	CD50	70.60 ± 0.26^{b}	9.17 ± 0.65^{b}	30.60 ± 0.10^{d}	11.19 ± 0.74^{g}	4.60 ± 0.14^{c}	6.18 ± 0.03^{a}	0.20 ± 0.10^{d}
	CD60	67.87 ± 0.23^{c}	8.30 ± 0.56^{c}	34.63 ± 0.12^{c}	16.09 ± 0.46^{e}	3.60 ± 0.14^{ab}	4.20±0.93^{c}	0.13 ± 0.06^{f}
	CD70	73.87 ± 0.97^{a}	7.67 ± 0.21^{d}	30.27 ± 0.15^{de}	12.55± 0.46^{f}	3.35 ± 0.21^{bc}	4.23±0.07^{c}	1.00 ± 0.20^{b}
	CD80	73.50 ± 1.06^{a}	9.15 ± 0.35^{b}	36.83 ± 0.81^{b}	20.91 ± 0.29^{d}	4.40 ± 0.85^{a}	6.15±0.21^{a}	0.57 ± 0.01^{c}
	CD90	52.40 ± 0.53^{d}	16.93 ± 0.49^{a}	38.17 ± 0.32^{a}	31.67 ± 0.64^{a}	3.90 ± 0.28^{ab}	5.30±0.05^{b}	1.43 ± 0.04^{a}
	VD50	46.47 ± 0.21^{g}	6.20 ± 0.58^{e}	27.03 ± 0.10^{g}	30.53 ± 0.37^{b}	3.27 ± 0.09^{cd}	3.56 ± 0.10^{b}	0.30 ± 0.03^{efg}
	VD60	48.03 ± 0.71^{ef}	6.40 ± 0.06^{e}	27.53 ± 0.36^{g}	29.11 ± 0.55^{c}	3.15 ± 0.03^{bc}	3.10±0.93^{e}	0.34 ± 0.06^{de}
	VD70	48.90 ± 0.62^{e}	6.56 ± 0.12^{e}	28.33 ± 0.30^{f}	28.47 ± 0.79^{c}	3.30 ± 0.05^{bc}	3.64±0.07^{cde}	0.57 ± 0.01^{c}
	VD80	49.03 ± 0.12^{e}	7.30 ± 0.12^{d}	29.73 ± 0.10^{e}	28.75 ± 0.35^{c}	3.46 ± 0.27^{bc}	3.72±0.21^{cd}	0.19 ± 0.01^{ef}
	VD90	47.37 ± 0.51^{g}	7.90 ± 0.21^{cd}	30.80 ± 0.32^{d}	30.71 ± 0.56^{b}	3.29 ± 0.04^{bc}	3.65±0.05^{cde}	0.43 ± 0.04^{cd}

Table 29 (Continued)

Samples	Drying condition	L*	a*	b*	ΔE	Ash content (%)	Protein content (%)	Fat content (%)
Lamella	FD	69.13 ± 0.35^{a}	5.37 ± 0.21^{g}	25.13 ± 0.06^{g}	3.77 ± 0.16 ^{e}	1.96 ± 0.03b	4.88 ±0.01^{f}	0.75 ± 0.05^{b}
	CD50	69.37 ± 0.25^{a}	8.45 ± 0.07^{d}	28.47 ± 0.23^{ef}	5.43 ± 0.16^{de}	3.65 ± 0.21^{d}	6.74 ± 0.08^{c}	0.47 ± 0.06^{c}
	CD60	67.67 ± 0.40^{b}	9.03 ± 0.46^{c}	33.67± 0.12^{b}	8.98 ± 0.51^{d}	3.05 ± 0.21^{a}	5.53±0.04^{c}	0.07 ± 0.30^{bc}
	CD70	69.83± 1.19^{a}	8.10± 0.52^{d}	31.40± 0.82^{c}	9.33 ± 0.49^{d}	3.05 ± 0.21^{a}	5.28±0.01^{e}	1.50 ± 0.10^{a}
	CD80	68.90± 0.10^{a}	9.63± 0.38^{b}	33.97± 1.36^{b}	22.25 ±1.41^{b}	3.25 ± 0.07^{a}	5.72± 0.11^{b}	0.50 ± 0.10^{bcd}
	CD90	55.63± 0.85^{c}	14.57± 0.31^{a}	36.50± 0.46^{a}	17.52 ± 0.10^{c}	2.65 ± 0.07^{ab}	6.11±0.004^{a}	1.67 ± 0.21^{a}
	VD50	44.17± 0.49^{f}	6.00± 0.32^{f}	27.26± 0.57^{f}	27.62 ± 2.70^{a}	2.57 ± 0.004^{cd}	5.47 ± 0.02^{d}	0.47 ± 0.04^{cd}
	VD60	46.90± 0.96^{e}	6.86± 0.46^{e}	28.33± 0.26^{ef}	25.68 ± 3.77^{ab}	2.94 ± 0.12^{ab}	5.43±0.02^{cd}	0.46 ± 0.06^{cd}
	VD70	49.80± 0.46^{d}	5.83± 0.10^{fg}	28.96± 0.21^{de}	22.71 ± 2.42^{b}	2.77 ± 0.03^{ab}	5.43±0.08^{cd}	0.36 ± 0.04^{d}
	VD80	50.63± 0.31^{d}	5.66± 0.15^{fg}	29.20± 0.06^{de}	21.97 ± 2.75^{b}	3.13 ± 0.02^{a}	5.32±0.05^{de}	0.51 ± 0.04^{bcd}
	VD90	47.67± 0.31^{e}	7.13± 0.17^{e}	29.90± 1.37^{d}	24.76 ± 2.76^{ab}	2.90 ± 0.10^{ab}	5.49±0.02^{c}	0.42 ± 0.03^{cd}

Table 29 (Continued)

Samples	Drying condition	L*	a*	b*
Pulp waste	FD	66.50 ± 0.17^{a}	6.87 ± 0.61^{fg}	28.00 ± 0.35^{e}
	CD50	44.47 ± 2.31^{d}	14.73 ± 0.29^{a}	32.13 ± 1.30^{c}
	CD60	51.97 ± 1.06^{c}	14.17 ± 0.49^{ab}	34.50 ± 0.53^{b}
	CD70	60.70 ± 1.21^{b}	13.60 ± 1.15^{b}	37.43 ± 0.40^{a}
	CD80	60.67 ± 1.33^{b}	10.53 ± 0.21^{c}	34.13 ± 0.35^{b}
	CD90	34.97 ± 1.33^{e}	14.23 ± 0.57^{ab}	25.03 ± 1.07^{f}
	VD50	43.60 ± 0.70^{d}	6.63 ± 0.20^{g}	28.26 ± 0.91^{e}
	VD60	45.37 ± 0.42^{d}	7.63 ± 0.10^{ef}	30.83 ± 0.10^{d}
	VD70	44.87 ± 0.25^{d}	8.36 ± 0.06^{de}	31.43 ± 0.36^{cd}
	VD80	45.20 ± 0.20^{d}	8.56 ± 0.15^{d}	31.76 ± 0.50^{cd}
	VD90	43.50 ± 0.66^{d}	9.03 ± 0.26^{d}	30.56 ± 0.15^{d}

Values are mean ± SD, significant different ($p < 0.05$) of data were represent with the small letter (a,b,c) within the column based on specific part of pome

Green color indicates the existence of chlorophyll in the flavedo which is fat soluble, in line with the highest fat content in flavedo (Table 29) compared to other parts of pomelo residues. This pigment is sensitive to heat and acid (Barrett et al., 2010) thus, leading to degradation of the pigment particularly during drying.

Consequently, the loss of chlorophyll leads to product becoming yellowish. The value of b* represents the yellowish color, the higher the b* value means the yellowish color is increasing. CD of flavedo showed that the b* value (34.6 - 39.3) to be significantly higher (p <0.05) than VD (27.0 - 29.5) in comparison with FD (36.9). Nevertheless, VD flavedo revealed insignificant deviation (p >0.05) at different drying temperature (50 - 90°C). The b value correlates in contrary trend with browning index (BI) value whereby lower BI value indicate bright yellow in color, and vice versa. These properties changed significantly depending on the drying methods. The CD flavedo (96.6 – 157.2) and VD flavedo (117.6 – 137.9) present significantly different (p < 0.05) BI value than FD flavedo (85.1) (Refer Appendix F; Table F1). Karaman et al. (2014) reports that the presence of polyphenoloxidase compounds that reacted with phenolics develop the browning pigment (higher BI value) during drying process.

In addition, the chroma (C) value which reflects the color intensity of flavedo can be refer to Appendix F (Table F1). The intensity of C was found to be significant (p < 0.05) and higher in CD flavedo (34.8 – 39.8) compared to VD (27.3 -30.0), but slightly similar with FD (37.0). Lower C value is shown by the brown color generated in the VD samples. The C values were correlates well with hue values. In terms of hue, (h*), overall findings produced value more than 80 except at higher temperature (90 °C) which only generated CD (67.7) and VD (79.5) respectively. A lower value of hue represents higher yellow appearance in the assay (Pathare et al., 2013). This indicates that the green color is fading, and yellow color of dried product is increasing which differing on drying temperature applied. Furthermore, insignificant difference (p > 0.05) of h* value of VD (80.54 – 81.44) and CD (84.90 - 81.71) was discovered at different drying temperature (50 – 80 °C) implies both drying method qualified to preserve the pigment(chlorophyll) in flavedo.

Lastly, the most significant parameter which could be a superior indicator is known as total color change (ΔE). FD flavedo appeared to be lowest ΔE value

compared to CD (14.0 - 23.6) and VD (28.2 – 32.4). Briefly, the E values were significantly ($p < 0.05$) influenced by vacuum drying more than conventional drying. This is likely to be related to the reduction of sugar content (glucose) which lead to occurrence of enzymatic reaction and Maillard product indicating greater changes of E for VD process. Different drying methods such as vacuum oven drying and conventional drying reveals lower glucose content (40.5g/kg and 48.3 g/kg, respectively) compared to FD(55 g/kg) (Karaman et al., 2013)

Post drying methods were evaluated to verify the changes of flavedo part of pomelo residue. Conventional and vacuum drying revealed significant ($p < 0.05$) diverse color properties after being treated at different drying temperature. Lower L*, b*, and hue values were obtained as drying temperature increased and the value of a*, chroma, BI and total color change increased (refer Appendix F; Table F1) under the same condition. However, insignificant variation ($p > 0.05$) were obtained in vacuum drying at different drying temperature compared with CD and this reflects the stability of related pigments is better than CD. At lower temperature (50 °C) CD flavedo displayed similarities in every aspect of color properties with FD. Overall, different thermal drying methods do affect the color properties in the current study.

4.2.2.2 Albedo

Focusing on albedo which are the whitish part of pomelo peels, it was found that FD albedo has L* value of 70.1, a* value of 3.4 and b* value of 24.6 as summarized in Table 4.3. However, after being treated at different drying temperature (50 – 90 °C), a decreasing trend was observed particularly during CD (70.6 - 52.4). The L value which represents brightness is closely related to the browning level of samples and the lower value of L means more browning reaction occurred. Meanwhile, consistent L value ($p > 0.05$) were found at similar range temperature (50 – 90 °C) of VD albedo (46.5 - 49.0) which parallel to the result of L (43.20 - 41.33) for pulsed vacuum drying reported by Wang et al. (2018) revealed insignificant value ($p > 0.05$) within the range 60, 65 and 70 °C.

Generally, with increase in drying temperature, L* decreased whereas a* and b* of albedo increased in both corresponding drying (CD and VD) methods. This is due to high temperature which accelerated the browning reaction of pomelo albedo

during drying process due to enzymatic and non-enzymatic reaction which occurred during processing (Nowacka et al., 2012).

The a* value of dried albedo increased significantly ($p < 0.05$) during CD (0.17-16.93), while no significant variation ($p > 0.05$) was observed during VD (6.20-7.90) at different drying temperature compared to FD (3.37). Similar trend was observed with previous study when high temperature (75 °C) was applied which resulted in significant effect ($p < 0.05$) on the color (3.44) compared to FD (- 0.82) of the lemon slices (Wang et al., 2018).

The findings on b* value under both drying methods (CD and VD) were significantly differed at different drying temperature. Enhanced b* value was observed in CD (30.27 - 38.17) and VD (27.03 – 30.80) albedo as drying temperature rises (50 – 90 °C) (Table 29) which reflects higher yellowish color of albedo compared to FD albedo (24.6). This reflects well with lower value L of VD albedo (48.90) and higher b value (28.33) at 70 °C comparing with FD albedo (70.13). Similar trend of lower L (35.80) and higher b value (3.44) was observed in pulsed vacuum dried of lemon slices at 70 °C than fresh (48.75) lemon slices (Wang et al.,2018;Bejar et al., 2011; Ghanem et al., 2012).

Hue is considered most visual color parameters compared to others (Bai et al., 2013). Hue value decreased from 73.3 to 66.1 for CD, but increasesd from 77.1 to 75.6 for VD at different drying temperature, which is lower than FD (82.21). This indicates that the lower hue value (H<90) will result in darker orange-red color. This correlates well with the value of chroma and browning index, which increases as temperature increase in both CD and VD methods.

Browning index (BI) of CD shows significant increase ($p < 0.05$) trend from 59 to 141, meanwhile for VD the value of BI was not significantly different ($p > 0.05$) from 92.81 to 110.25 from low to high drying temperature. The higher value of BI is reflected in the darker brown color as observed in the present study. This indicates the instability of color pigments in albedo or melanin compounds (brown products) generated during condensation or polymerization of o-quinones during drying. In addition, these appeared due to the fact that the CD included oxygen exposure and

long drying time (24 hr) creates an adverse biochemical reaction (Karaman et al., 2014; Xie et al., 2017; Riva et al., 2005; Chong et al., 2005).

The total color change is often used to evaluate the magnitude of overall color differences between dried and control. The differences of total color change can be classified as very distinct ($\Delta E>3$) distinct ($1.5<\Delta E<3$) and small differences ($\Delta E<1.5$) (Adelakun et al., 2009). The results show total color differences of dried pomelo albedo compared to control which indicates that temperature and drying method significantly ($p < 0.05$) affects the total color change. Total color change (ΔE) for FD is only 7.6 which showed lesser value than the average obtained from CD (11.2- 31.7) and VD (28.5 - 30.71) methods. Even though the overall value of ΔE showed VD is slightly higher than CD, no significant variation ($p > 0.05$) was observed at different drying temperature, compared to CD method. Overall, VD shows similar or minimal effect on pomelo albedo in terms of color in comparison to CD methods.

4.2.2.3 Lamella

For lamella, the color properties are affected by different drying methods at different drying temperature and the figures are displayed and summarized in Table 29. Dehydration process result in significant changes on the color of dried lamella. Post drying refers to after drying process appeared to be one of the apparent process that significantly ($p < 0.05$) affect L*, a*, b*, h*, C*, BI and E values compared to control (FD).

Particularly, different drying methods revealed varied figures in L value which are within the range of 55.6 – 69.4 for CD, while for VD the values lie between 44.2 – 50.6 compared to FD (69.1) lamella. However, no significant difference ($p > 0.05$) were obtained between CD dried at 50 ℃ (67.7) and FD (69.1). In addition, significant changes ($p < 0.05$) of a, b and chroma value as drying temperature is increased in both drying methods (CD and VD) correspondingly.

In its natural condition lamella has a slightly pinkish color in certain part, which attributable to the presence of lycopene. This pigment is sensitive to heat and degrade accordingly when heat is applied (Xu et al., 2007). The h value reduced significantly ($p < 0.05$) when higher temperature is applied. Nevertheless, VD lamella

at 50°C (77.6), 70 °C (78.6) and 80 °C (79.0) showed no significant variation ($p > 0.05$) with FD lamella (78.0).

During the drying process, browning index were found to be significantly affected ($p < 0.05$) by CD (61.1 - 118.8) and VD (91.51 – 103.8) in time compared to FD (50.0). This can be explained by the presence of heat sensitive compounds, such as proteins (8.18 – 10.47% in current study) and carbohydrates that adjusted the color of dried fruits. Consequently, the total color change was evaluated and were found that the color parameters correspond and were inter-related with each other (Miranda et al., 2009). For instance, at higher temperature (90 °C) value of L* (55.6) lamella was lower while higher value of a* and b* were generated at temperature (50 – 80 °C). Most literature reviews briefly explain about the color of fresh fruits which includes the flavedo part and the juices extracted but limited information can be found on impact of drying on the color of lamella parts. Thus, E value of dried lamella were significantly influenced ($p < 0.05$) at different drying temperature and different drying methods. The average value of maximum E value found in VD (27.6) at 50 °C are greater by 9 times more than CD (22.3) at 80 °C which only increased by 7 times compared to FD (3.8). However, E values during vacuum drying of lamella at different drying temperature were found to be not significantly different ($p > 0.05$) with each other. In general, the FD process has minimum effect on color changes and quality which reflects more on fresh fruits compared to CD and VD in the current study.

4.2.2.4 Pulp waste

The color, browning index and total color change of dried pomelo pomace at different drying temperature for CD and VD are presented in Table 29. Color denoted by CIE lightness (L, a, b) and browning index (BI) of conventional drying and vacuum drying process were different significantly ($p < 0.05$).

At higher temperature, the hue angle reduced significantly ($p < 0.05$). It should be pointed out the hue angle was in the range of < 90 that corresponds to yellow-orange-red (brown) color. The lower hue angle value indicates the darker color of the material. The chroma level increases significantly ($p < 0.05$) during high drying temperature (Appendix F, Table F1). This indicates the instability and degradation of the color pigment when dried at a higher temperature (Chong et al., 2008; Lee et al.,

2012). Consequently, the findings of total color change of CD revealed that the value increased compared to vacuum drying in overall temperature. This reveals acceleration of Maillard and enzymatic reactions take place during CD compared to VD that led to formation of brown pigments (Kiranoudis et al., 1993; Michalska et al., 2016). Furthermore, caramelization process occurred during higher drying temperature (Ghanem et al., 2012; Ruíz Díaz et al., 2003) is likely to be one of the reason. A similar trend was observed by a study by Wang et al. (2018), where they found that total color change of dried lemon slices were significantly increased ($p < 0.05$) using pulsed vacuum drying process during increasing drying temperature (60, 70 and 75°C). No significant changes ($p > 0.05$) were observed for total color change particularly VD pomelo pomace at 70, 80 and 90°C compared to FD pomelo pomace.

Generally, higher drying temperature, particularly in conventional drying does affect the color significantly ($p < 0.05$) in overall parts of pomelo residues. In contrast, different drying temperature during VD did not affect the color properties of pomelo residues significantly ($p > 0.05$). In terms of different drying methods, CD processing does affect the color significantly ($p < 0.05$) in particular temperature. Instability of color compound were observed in conventional drying condition compared to vacuum drying based on overall properties. Nevertheless, significant variation ($p < 0.05$) of results produced depends on condition of drying methods and parts of pomelo residues accordingly. Freeze drying displays color stability during dehydration process as the sublimation process was applied at low temperature (-45°C), while at the same time preserving the pigment from being destroyed (Rafiq et al., 2019).

4. 2. 3 Effects of drying methods and temperature on nutritional composition

4.2.3.1 Ash content

Ash content indicates the inorganic material present particularly content of vitamins and mineral. It is one of the significant elements to identify the quality of functional foods (Hofman et al., 2002). The mean values of different drying methods on proximate composition for the pomelo residues are summarized in Table

29. In summary, the results of ash content is within the range from 5.9 to 7.15 % DW for flavedo, from 2.64 to 4.4% for albedo, from 1.96 to 3.25% for lamella, and from 10.65 to 3.14 % for pulp waste. In term of freeze-dried pomelo residues, the highest ash amount is present in flavedo (5.9 %) followed by pulp waste (3.24 %) albedo (2.64 %) and lamella (1.96%). Higher nutritious value is found on outermost part of fruits through inward part of the flesh (Abdullah, et al., 2012) due to the existing colored pigment were conferred by phytochemical attracting pollinators for the seed dispersal.

For the outer part or exocarp of the peels, CD for flavedo showed a range of ash content between 6.5 - 7.15 % and VD at a range of 6.29 - 6.52% which is higher than ash content found in the FD (5.92 %). Significant increase ($p < 0.05$) of ash content (Refer to Appendix D, Table D6) was observed during CD and VD in comparison with FD. It might be due to the elevated temperature, when the MC reduced drastically, which correlates to removal of moisture, and the remaining dried flavedo only consisted of dried matter (without moisture) (Rafiq et al., 2019). This may reflect higher value of ash content in the dried flavedo (Nollet & Toldra, 2015) compared to freeze dried flavedo. In addition, the lower the MC of pomelo peels, the greater value of ash content recorded. For example, consistent with the MC of CD flavedo at 90 °C (4.85%) showed lower MC (Table 28) than FD flavedo (9.33%) reflects the higher drying matter in CD more than FD flavedo that tend to demonstrated higher ash content respectively. Previous research recorded a similar trend (Naidu et al., 2015; Nollet & Toldra, 2015) revealing the existence of lower value of inorganic nutrients after oven dried (1.88%) and vacuum dried of persimmon powders (1.88%) compared to fresh ones (0.35%) (Ghanem et al., 2012; Karaman et al., 2014). In addition, similar results were obtained by Rafiq et al. (2019) which reported tray-dried Kinnow peels contained higher (0.57%) value than the fresh ones (0.49%). This can be explained by the convective heat transfer mechanism in tray drying which could cause more increment in the solubility of the minerals (Arslan and Ozcan, 2008).

The results of CD flavedo (6.45 – 7.15%) are comparable with citrus peels (5.15 – 6.43%) as presented by Abirami et al. (2014). However, the results are insignificant ($p > 0.05$) during post drying (CD and VD) at different drying temperature. In contrast, the results of the dried flavedo is significantly lower ($p < 0.05$) than fresh

flavedo and the results are similar to those obtained by Abirami et al. (2014) and Karaman et al. (2014). Thus, it can be concluded that CD is a better method for drying to retain vitamin and mineral in flavedo, followed by VD and FD.

The ash content value for albedo (FD) was recorded at 2.64%, for CD it is between 3.6% to 4.6% and for VD it is between 3.15% to 3.46% during drying temperature at 50 – 90 °C. The findings in Table 4.7, indicate the ash content increase by more than 20% on average in CD and VD at different drying temperature. There is no significant differences ($p > 0.05$) of FD with VD at different drying temperature (50°C - 90 °C). Meanwhile, CD exhibits significant improvements ($p < 0.05$) on ash content compared to FD at different drying temperature. The ash content of CD albedo (3.6 to 4.6%) is comparable with the previous study of dried pomelo fiber (3.41%) (Mat Zain et al., 2014) and the value of 2.94% - 3.03% for dried orange fiber (Crizel et al., 2014; Ghanem et al., 2012). In general, post drying of albedo do not significantly affect ($p > 0.05$) the ash content at different drying temperature.

Whereas for lamella, the ash contents from FD, CD and VD were found to be 2.0%, 2.7 - 3.7% and 2.6 - 3.0% respectively. The results of CD lamella (2.7% - 3.7%) are found to be insignificant ($p > 0.05$) compared to FD lamella, but are significantly different ($p < 0.05$) with fresh lamella (0.87%). These might be due to the presence of the remaining dry weight (Table 28) during post drying on respective lamella. For instance, FD lamella displayed lower MC than CD lamella, resulting in lower ash content in FD lamella compared to CD lamella, which could be due to spattering process (Nollet and Toldra, 2015) during incineration phase which could decrease the volume and ash content. There is significant difference ($p < 0.05$) value of ash content obtained by VD lamella at different drying temperature although limited studies related to lamella have been discussed and reported. However, the ash content of CD lamella was comparable with the pomelo peel fiber (3.17% -3.82%) as reported by Abirami et al. (2014).

For the remaining pomace after juice extraction, the pulp waste consists of ash content within the range from 4.0% to 10.7% for CD, from 3.2% to 3.5% for VD in comparison with FD (3.2%). CD pulp waste which dried at 50 °C (10.7%) exhibited significantly ($p < 0.05$) higher ash content compared to FD (3.2%) and VD (3.2% - 3.5%)

at different drying temperature. Conventional drying appeared to have improved ash content more than 25% content in comparison with VD which improved by less than 10%.

Results of the current study showed higher ash content in comparison to previous study on dried peel, dried pulp and dried peel fibre (3.2 - 6.4%) (Abirami et al., 2014). In addition, comparable results (~2.6%) can be obtained with dried orange peel and pulp by-product flour which has been dried at similar temperature (Nassar et al., 2008), and dried orange peel fibre (2.9 % – 3.0%). The reason for the higher ash content value could be due to the presence of heavy amounts of inorganic nutrients (potassium, sodium, calcium, phosphorus and magnesium) in the pulp waste (Czech et al., 2020; Aletor et al., 2002; Iqbal, et al., 2012) is likely to be related to the remaining juice retained in the pulp waste after being extracted.

Ash content indicates the presence of vitamin and total mineral compound in pomelo residues. It is needed by humans to be in ideal health. Overall, post drying effects the total mineral compound and mineral content. Thus, it is important to identify the effects of processing (drying) on ash content and select the best condition to retain the amount after processing. In general, both drying methods improved ash content of pomelo residues significantly ($p < 0.05$). In comparison the effects of retention value of ash content during drying process, the current findings of ash content from maximum value to minimum value are as follows: CD > VD > FD. In term of the effects of different drying temperature, no significant ($p > 0.05$) difference is observed at 50 – 90 °C.

4.2.3.2 Fat content

Fat content is significant to maintain the texture, flavor and pigments of fruits (Shamsudin et al., 2009). The findings of the effects of different drying methods on fat content of pomelo residues at different drying temperature is summarized in Table 29.

In brief, the findings indicate FD flavedo exhibits higher fat content (1.6%) followed by pulp waste (1.1%), lamella (0.75%) and albedo (0.6%) respectively. This can be explained by the large presence of oil glands that found in leaves, stems, flowers and fruits particularly located in the outermost part of the rind. Essential oils

(0.172 - 0.185%) have been reported varies in fresh pomelo of Thailand cultivars (KP, KY) (Chaiyana et al., 2014). After being dried with CD (0.7% - 1.3%) and VD (0.6% - 1.3%) processes, the fat content reduced significantly ($p < 0.05$) in comparison with FD (1.63%). This is due to sustained structure of tightly packed parenchymatic cells (Thielen, et al., 2013) in flavedo which leads to higher fat content during FD. The CD flavedo and VD flavedo exhibited lower fat content by more than 20% compared to FD, which also affected the texture, color, flavor, aroma and nutritive value. Lower temperature treated for FD flavedo released a better aroma (Vega-Gálvez et al., 2009; Karaman et al., 2014) due to limited exposure on oxygen which could retain the essential oil in flavedo, and comparable results were obtained by Karaman et al. (2014) as well. Among different parts of pomelo residues, flavedo has higher fat content than other parts, however, it has lower fat content than the dried peel (3.6% - 4.1%), dried citrus pulp (5.2% - 5.5%) and dried peel fibres (2.8% - 3.0 %) (Abirami et al., 2014). In terms of different drying temperature (50 – 90 °C), the results showed no significant difference ($p > 0.05$) in both CD and VD.

The albedo with the thickest and whitish part of pomelo peels possessed the lowest fat content (0.6%) compared to other pomelo residues (0.8% - 1.6%). This result can be explained by the fact that albedo comprised air-filled intercellular spaces (Thielen et al., 2013;Crizel et al., 2014) and they are highly porous, that leads to easier loss of essential oils. and this characteristic is agreed by previous research (Chaiyana et al., 2014).

In terms of different drying methods, significant variations ($p < 0.05$) of findings were obtained in CD albedo at 50 – 90 °C compared to drying in VD process, which did not show variations at certain temperature (50 °C ,60 °C, 90 °C). Conversely, at higher drying temperature (70 °C and 90 °C) fat content was found to be significantly ($p < 0.05$) higher (1.0% and 1.43% respectively) compared to other drying temperature. One of the reason is due to firm layer formation on outer part of albedo during conventional drying which eventually limits the loss of fat. In other words, the fat-water emulsion could be broken down easily during high temperature, which leads the fat to dissolve easily in the organic solvents, facilitating the free fat bound from the tissues of the foods (Nawar, 1996; Nielsen, 2010), to be retained inside the albedo.

In contrast, during vacuum drying (0.43%), the fat content was lower than CD (1.43%) at 90 ℃ which due to the presence of fat which combined together with higher proteins content during CD (5.30%) than VD (3.65%) respectively. In vacuum condition, eventually it increase the boiling point of water at lower pressure. Higher rate of moisture loss leads to accumulation of dried material which consists of fat, protein and minerals. Nevertheless, bound fat with protein and carbohydrate, are not easily extracted with organic solvents as claimed by Nielsen (2010). Thus, lower value of fat content was generated during vacuum drying. This is consistent with Kinnow peels; citrus fruits after being dried result in non-significant decrease in fat content that might be due to exudation of fat with moisture evaporation (Rafiq et al., 2019).

Peels known as lamella are the thin layer which cover the pulp of juice sac, comprise fat content of FD (0.8%), within the range from 0.5% to 1.7% for CD and from 0.4% to 0.5% for CD in general. Equivalent with conventional dried albedo, the higher temperature (70℃ and 90 ℃) applied result in higher fat content (1.5 % and 1.7% respectively) retention in CD lamella. Likewise, with previous study revealed the result obtained by orange peels (2.0%) and Mandarin peels (2.2%) based on CD at 70 ℃ (Marey and Shoughy, 2016). Naturally, the seeds were attached inside lamella together with the pulp. The presence of seeds that attached together with lamella during drying process leads to higher value of fat content occurring during analysis.

Nevertheless, at lower temperature (50 ℃ and 60 ℃) significant decrease ($p < 0.05$) of fat content (0.5% and 0.07%) was observed in conventional drying, meanwhile, similar trend appeared in vacuum drying as well except that the lower fat content was recorded at all drying temperatures in comparison with FD (0.8%). However, at different drying temperatures (50 ℃ - 90 ℃), no significant variation ($p > 0.05$) of fat content lamella was observed during vacuum drying. However, few studies have reported on effects of drying on dried lamella parts of citrus fruits particularly for vacuum drying method.

Subsequently, pulp waste which are pomace remaining after juice extraction, the fat content recorded was 1.1% (FD), from 0.6% to 1.4% (CD) and from 0.2% to 1.0% (VD). As pulp waste are dried, it becomes flakes and the value of fat content was 1.1% which was slightly higher than lamella (0.8%) and albedo (0.6%) but

value of fat content is lower compared to flavedo (1.67%) during FD. This can be explained by presence of major compound (limonene is a major component in oil of citrus peels) from seeds remained in pulp waste after juice extraction process (pulp waste) (refer section 3.2) resulting higher fat content. This is supported by the presence of limonene content from seeds (2615.30 ppm) was higher than the peels (169.30 - 265.14ppm) (Pichaiyongvongdee & Haruenkit (2009) .The results are supported by previous studies whereby citrus pulp (5.23 – 5.45%) of *C. hystrix* and *C. maxima* (red and white) contains higher fat content compared to the peels (Abirami et al., 2014). However, the fat content of dried pulp waste decreased at lower drying temperature (50 °C and 60 °C) revealed 0.73% and 0.60% correspondingly by more than 30% compared to FD (1.07%). This can be explained by oxidation of fat into other compounds and exuded along with moisture evaporation process (Wu & Mao, 2008; Akonor et al., 2016).

Similar trend was recorded with VD pulp waste at different drying temperature whereby the drying temperature does not have a significant influence ($p > 0.05$) on fat content during VD and FD. This could be attributed to the absence of oxygen under vacuum condition, limiting the oxidation of fat from occurring which leads to minimum changes during different drying temperature. On the other hand, the present study observed that fat content from pulp waste (< ~1.2%) was found to be lower than observations from previous study in *C. hystrix, C. maxima* which showed the fat content to be within the range of 2.8% and 5.45%, while orange by-product showed 4.4% (Abirami et al., 2014; Fernández-López et al., 2009). Unique variation of cultivar differed significantly based on different factors such as growing region, nutritional qualities, and cultural practices.

In general, fat content reflects the refreshing flavors, as well as the sweet and delicious taste of the fruits. As it is commonly present at the exocarp of the fruits other than stems, leaves and flowers (Jena et al., 2009; Chaiyana et al., 2014), it is important to identify the effects of drying temperature and different drying methods on the pomelo residues. Thus, the findings of the current study was found to be similar with findings from previous research where the highest fat content is exhibited by

flavedo followed by pulp waste, lamella and albedo respectively. In terms of different drying temperature applied, there was no significant effect ($p > 0.05$) on fat content particularly in VD. Significant consequence ($p < 0.05$) was observed when different drying methods (CD and VD) are applied on pomelo residues. Thus, CD was assumed to be a better drying method due to the insignificant ($p > 0.05$) effect on the fat content than VD in comparison with FD.

4.2.3.3 Protein content

Protein is one of the important macronutrients needed by human beings to safeguard and maintain the cell functions and growth efficiently. Table 29 presents the effects of drying methods and drying temperature on protein content of pomelo residues.

The protein content of pomelo residues was evaluated for the control group (FD). Among pomelo residues, protein content was found to be the highest in pulp waste (8.2%), followed by flavedo (7.6%), lamella (4.9%) and albedo (3.5%). Pulp waste was also found to have a significantly higher ($p < 0.05$) protein content compared to other parts of pomelo residues. This is believed to occur due to the presence of nitrogen component in the juice which remained during post drying and therefore, contributes to higher value of protein content. Proteins composed of the chain of amino acids linked by peptide bonds includes hydrogen, carbon, nitrogen, oxygen and sulfur. Nitrogen is the most distinctive element present in proteins. In brief, protein rich in basic amino acids contain more nitrogen (Nielsen, 2010). Total organic nitrogen are primarily from proteins and may include all organic nitrogen-containing non-protein substances (S´anchez-Moreno et al., 2006).

Next, focusing on flavedo (exocarp of pomelo fruits), this study investigated the protein content affected by different drying methods at different drying temperature. The protein content of flavedo showed significant difference ($p < 0.05$) among different drying methods. For instance, CD flavedo showed protein content values within the range of 10.7 to 11.6%, while for VD flavedo the protein content values were between 8.6% to 9.2% in comparison with FD flavedo (7.6%). In view of the results obtained, the percentage improved significantly ($p < 0.05$) in CD by

52% (maximum value) compared to VD 20% at a particular temperature. Higher protein content in both drying method (CD and VD) compared to FD, reflects that the different drying operation does affect the protein content.

As for different drying temperature, increased temperature that occurred in CD, the protein content of flavedo tend to increase as well, which is likely due to increase the rate of degradation on the peptide bond between the chain of amino acids and subsequently leads to higher nitrogen (protein) compound measurement. In contrast, it is apparent that protein content decrease ($p < 0.05$) with increasing drying temperature during VD. This might be due to the rate of moisture loss was lower in VD compared to CD (Table 28) flavedo, in other words, conjugated protein which also contain non-amino acid components still remained in VD flavedo, thus, the exact protein content available is limited. The reduction of protein content after drying due to Maillard reaction as it causes changes in food composition between protein and available carbohydrates. Furthermore, formation of complexes between anti-nutritional components and protein in the presence of heat treatment might contribute to reduce the probability of available protein (Enomfon-Akpan & Amoh, 2004; Lee Hoon Ho et al., 2016; Rafiq et al., 2019).

As the outermost part of pomelo peels (except of pulp waste) contains the highest protein content (7.63%) , this parallel with previous studies which reported that higher nutritious value are located on outermost part of fruits through inward part of the flesh (Abdullah et al., 2012).

In addition, the protein content of dried flavedo and pulp waste were higher than the dried peel fiber from citrus samples (7.51–7.83%), dried peel and pulp by-products of orange (4.75–5.15%), dried orange fiber (8.50–8.93%), dried Fino lemon (7.92%) and dried lime residues (8.08– 8.24%) (Abirami et al., 2014; Crizel et al., 2013; Peerajit et al., 2012; Nassar et al., 2008; Figuerola et al., 2005). In general, CD showed better retention of protein content after drying compared to VD and FD.

The whitish part of pomelo residues, the albedo, was observed to have protein content within the range of 4.2% to 6.2% for CD, from 3.1% to 3.7% for VD in comparison to FD (3.5%). However, no significant difference ($p > 0.05$) was found on the protein content in VD albedo in comparison with the control group. Despite similar

trend was observed in flavedo, maximum percentage of retention on protein content were found to be more than 50% in CD compared to VD (only <7%) particularly at 50 °C and 80 °C. Comparable finding was obtained by previous research on fiber concentrate from dried Valencia orange (6.7%) and dried Eureka lemon (6.8%) (Figuerola et al., 2005). In addition, no significant variation ($p > 0.05$) were displayed between different drying temperatures.

Lamella contains different protein content value after being treated by different drying methods. The findings of protein content were between the ranges of 5.3 to 6.7% for CD, from 5.3 to 5.5% for VD compared to FD lamella (4.9%). It can be seen that the percentage of protein content appears to decrease as drying temperature from 50 °C to 70 °C is applied during CD, which might be associated with the loss of protein due to denaturation or changes in solubility during drying (Miranda et al., 2009). Similar trend was observed during VD, however, insignificant variation ($p > 0.05$) of protein content (5.32 - 5.47%) was observed at different drying temperature. In general, crude protein was not affected perhaps due to the protective action that certain sugar confers which eventually preserved the native protein structure upon loss of water (Issis et al., 2019). Comparable findings (6.07%) was observed on previous research obtained by citrus waste (Pourbafrani et al., 2010; Negro et al., 2016).

The pulp waste possesses protein content which are comparable with flavedo parts of pomelo fruits. Similar trend is observed with dried flavedo, albedo and lamella whereby conventional and vacuum drying significantly increased ($p < 0.05$) the protein content of pulp waste. The results vary depending on drying methods involved for instance, from 8.3% to 10.5% for CD, from 9.1% to 9.5% for VD while FD obtained 8.2% protein content. It can be seen that low drying temperature during CD had the greatest influence on protein content. Nevertheless, at 70 °C drying temperature, it is apparent that reduced protein content, which is expected to be due to amino acid released from the native protein denaturation during heating process reacted with other compounds like sugars to produce dark brown-colored polymers, called melanoidins via Maillard reaction process (Lee & Shibamoto, 2002; Pereira, 2005; Miranda et al, 2009). As for VD methods, insignificant difference ($p > 0.05$) can be

observed at different drying temperatures, whereby the protein content was higher than protein content in control (FD).

In general, different drying methods were found to ($p < 0.05$) affect the protein content in pomelo residues significantly. CD influenced more than VD in retaining the protein content for overall parts of pomelo residues. Thus, different parts of pomelo residues also varied with the highest protein content in pulp waste, followed by flavedo, lamella and albedo.

4. 2. 4 Effects of drying methods and temperature on total phenolic content

The variation of total phenolic content with different drying methods for different parts of pomelo residues at different drying temperature is shown in Figure 9.

4.2.4.1 Flavedo

The finding of the present study indicates that the flavedo, the exocarp part of pomelo peel, exhibited TPC content in range from 1225.69 - 3016.27 mg GAE/100 g DM for conventional drying (CD), whereas for vacuum drying (VD) the range was within 1281.82 – 1948.26 mg GAE/100 g DM) and followed by freeze-drying(FD) (1045.14 mg GAE/100 g DM). FD acts as a control in the current study. Slight improvement by 17% and 12% after drying take place at 50 and 60 °C respectively compared to FD. Even though, no significant variation ($p > 0.05$) of TPC value can be observed at 60°C during CD, but CD and VD flavedo showed a significant improvement ($p < 0.05$) of TPC content in comparison with FD.

The development of the TPC value is in line with the values in previous research done by Sultana et al. (2012). Increase of TPC in oven dried apricot was detected as the high molecular weight (bound phenolic) could be exposed to their free state when treated with heat treatment.

Nevertheless, the FD flavedo (1045 mg GAE/100 g DM) of the present study present similar results with bound extract from white Tambun pomelo peels (1595 mg GAE/100 g DW) (Lim & Loh, 2016) and shaddock peels (3400 mg GAE/100 g DW) (Oboh and Ademosun, 2011).

In contrast, the current study showed 4 times lower amount of TPC (9.48mg GAE/g FW) in comparison with previous research reported by Chang and Azrina (2017) which observed relatively higher TPC (38.67 mg GAE/g FW) after being treated with freeze drying process (Refer to Appendix D, Table D11). The variation in the TPC is likely to be due to changes of variety in plant cultivar as it can be influenced by genetic factors, postharvest condition, cultural practices and maturity stages (Odriozola-Serrano et al., 2008; Mäkynen et al., 2013; Xi et al., 2014). In contrast, previous studies reported on maqui; citrus fruits dried using vacuum oven revealed that vacuum drying condition was able to maintain these antioxidant compounds, which might be due to absence of oxygen, and shorter drying time at higher temperature (Issis et al., 2019).

4.2.4.2 Albedo

Findings showed TPC content for albedo is between range 1330 - 2926 mg GAE/ 100 g DM for conventional drying (CD) followed by vacuum drying (VD) (1791 - 2608 mg GAE/ 100 g DM) which were higher than treated with freeze drying (FD)(1268 mg GAE/ 100 g DM). As illustrated in Figure 9, the content of TPC after drying ranked from maximum value retained to least retained value are VD > CD> FD respectively. FD is used as a control in the present study. TPC content of CD was compared with FD. The results appeared to have improved by 4 - 5% at lower temperature (50 and 60 °C). Nevertheless, there is no significant difference ($p > 0.05$) in value of TPC at lower drying temperature of 50 and 60 °C. It increased by more than 50% as drying temperature increased (70, 80, 90°C). Meanwhile, the retention of TPC during VD improved more than 40% at lower temperature (50 – 60 °C). Greater improvement (more than 60%) of TPC was discovered at high temperatures (70, 80 and 90 °C).

Both corresponding drying (CD and VD) methods (1330.33 - 2608.42 mg GAE/100g DW) significantly improved ($p < 0.05$) TPC content as temperature increased, particularly at a higher temperature (90 °C) in comparison to FD (1267.87 mg GAE/100g DW) method. Chaaban et al. (2017) suggested that at higher temperature (100°C), non-enzymatic reaction could occur and produce new compounds. The new compounds activate inter-conversion either between phenolic-phenolic and other molecules (Que et al., 2008; M'hiri, et al., 2017

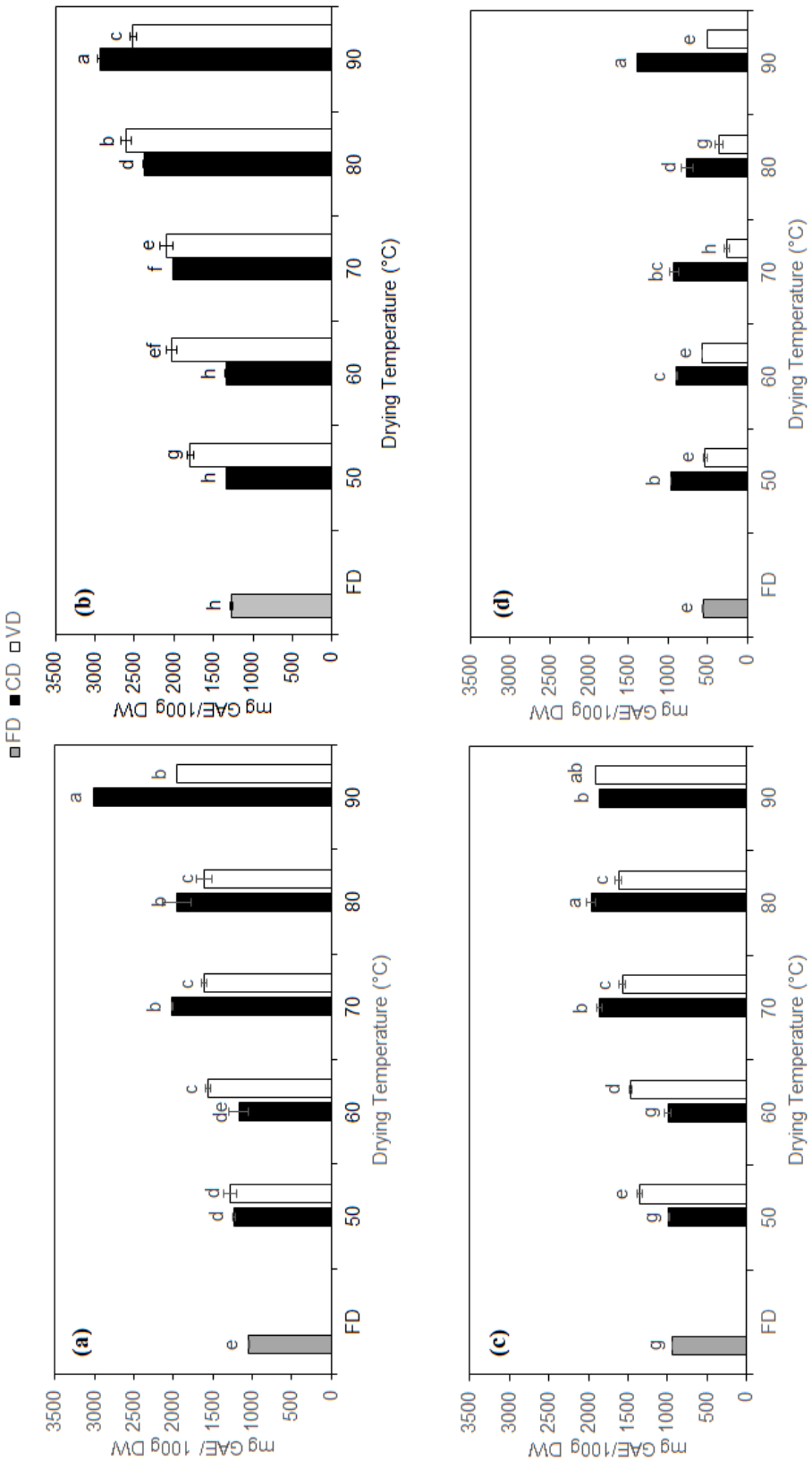

Figure 9 TPC affected by freeze drying (FD), conventional drying (CD) and vacuum drying (VD) methods of pomelo residues namely (a) flavedo, (b) albedo, (c) lamella, (d) pulp waste at different drying temperature (50-90°C).

In addition, drying processing step helps destroy the cell wall and allows phenolic compounds to be released from the insoluble portion of albedo, which can lead to increased level of TPC (Xu et al., 2017). A similar result was reported by Ding et al. (2017) which found an increase (559.6 – 577.5mg/100g DW) retention ratio of non-extractable phenolic (NEP) acid from lemon slices when the temperature (50 - 90°C) increased using air drying method. Nobiletin (3.2 – 4.0 mg /100g DW) and syringic acid from NEP of lemon slices showed an increasing trend as drying temperature increased from 50 - 90° C.

It was apparent that lower value of TPC content of FD albedo compared to CD and VD of albedo could be due to longer drying time that eventually lead to slower drying rate as this might contribute to critical condition for phenol composition extraction that will damage the compounds (Anagnostopoulou, et al., 2003; Geankoplis, 2003).

The CD of albedo (1330 - 2926 mg GAE/ 100 g DM) at different drying temperature (50 – 90 °C) was found to be statistically significant ($p > 0.05$) in comparison with FD albedo (1267.87 mg GAE/100g DM). A similar trend was reported when TPC of non-extractable lime wastes were dried using conventional oven at 60 °C (21.59 mg EAG/g DM) was slightly higher than after freeze drying treatment (21.15 mg EAG/g DM). Processing treatment, as well as environmental factors such as temperature and light intensity, could also affect the phenolic contents (Ioannou et al., 2018; Sun et al., 2015). Researchers discovered that by comparing the effects of both thermal drying method (CD and VD) on TPC, higher retention of TPC was obtained from VD (60%) in comparison to CD (5%) at 60 °C. Drying modes or operation affected TPC directly particularly if high temperature was used together with a long period of exposure to oxygen (M'hiri et al., 2017). Vacuum drying operation created a vacuum condition which limits the exposure of product to oxygen which could be the reason of higher retention of TPC. Previous researchers indicate that flavonoids are sensitive to oxygen leading to product degradation (Ramešová et al., 2012; Zenkevich et al., 2007). In brief, the current study observed that vacuum drying is better drying method for albedo to preserve TPC content.

4.2.4.3 Lamella

Lamella which covers the pulp, exhibits TPC content from VD in the range of 1354.58 – 1917.11 mg GAE/100 g DM, whereas CD resulted in TPC content between the range of 986.46 -1860.43 mg GAE/100 g DM followed by FD (942.49 mg GAE/100 g DM). The results indicate that TPC content increased by 4% – 108% while for VD the percentage increased by 44% -103% after a range of different drying temperature (50 - 90°C) was applied. However, no significant variation ($p > 0.05$) of TPC content was found during CD at lower temperature (50 and 60 °C) when compared with FD. In contrast, significant effect ($p < 0.05$) was observed between both drying treatments with freeze drying process of lamella. This indicates that high TPC of lamella can be the result of exposure to higher temperature that may cause the development of phenolic compound (low molecular weight) leading to increased antioxidant activities of heated citrus peels (Jeong et al., 2004). This is associated with the heat energy supplied during drying which is capable of breaking down the molecular structure of the covalent complex and releasing antioxidant compounds such as flavonoids, carotenes, tannins, flavoproteins and polyphenols polymers (Orikasa et al., 2014; Uribe et al., 2016).

The finding of FD lamella's TPC in present study (8.26 mg GAE/g FW) is different from previous research which reported the lamella part contained 30.66 mg GAE/g FW after freeze drying process. Variation of results in TPC of lamella, may be due to different varieties involved (Ledang varieties). (Chang and Azrina, 2017). Nevertheless, the peels of pomelo in the present study contained higher TPC compared to the peels of Tambun White and Pink pomelo which consists of 4.07 mg GAE/g FW and 3.01 mg GAE/g FW correspondingly. The presence of phenolic compounds predominantly varies among different types of fruits, geographical location, cultivation conditions, the structure of tissues and cultivar (Mac Dougall, 2002; Kumar et al., 2019).

4.2.4.4 Pulp waste

Pulp waste is obtained during post-extraction and they are the pulp that remained. In other words, the wastes (pulp waste) was collected after the juice was extracted from the fruits. In the current study, TPC content obtained in pulp waste

is within the range of 961.92-1387 mg GAE/ 100 g DM for CD, meanwhile for VD (254.20 - 569.66 mg / 100 g DM) followed by FD (554.65 GAE/100 g DM). Under conventional drying, pulp waste exhibits significantly higher ($p < 0.05$) TPC (762.36 - 1387.42 mg GAE/100g DW) compared to vacuum drying process (254.20- 569.66 mg GAE/100g DW) and freeze (554.65 mg GAE/100g DW) drying.

Greater value of TPC might be due to the exposure of oxygen in convection oven which leads to formation of Maillard reaction product and yielding the new phenolic compound from their precursor (Sultana et al., 2012) compared to vacuum drying process. It is consistent with the previous findings, from lemon pomace extracts which reported that the hot air method produced higher TPC than vacuum drying process (Papoutsis et al., 2017). Synthesis of gallic acid also can be attributed by the presence of oxygen in CD process which leads to higher TPC value (Papoutsis et al., 2017). Nevertheless, little information is available on the biosynthesis pathway of gallic acid (Vogt, 2010; Papoutsis et al., 2017). Generally, it can be indicated by the percentage of TPC rising more than 50% when treated at lower drying temperature (50 - 60 °C) during conventional drying process and at higher temperature (70 – 90 °C), the percentage of TPC rises by 67% - 150% significantly ($p < 0.05$) using the same drying method. This increasing trend is likely to be related to the occurrence of non-enzymatic reaction such as Maillard reaction and caramelization which end up forming free phenolic compound when high temperature is applied (Chen, et al., 2009; Liu, et al. 2007; Nicoli et al., 1999), where eventually, the result of TPC would become higher. Nevertheless, VD pulp waste (529.48mg GAE/ 100g DW) produced insignificant value ($p > 0.05$) in comparison with freeze dried product (554.65 mg GAE/100g DW).

In the present study based on the results of freeze drying of pomelo residues, pulp waste was found to consist the lowest amount of TPC (554. 65mg GAE/100DW) compared to other peels (flavedo, albedo and lamella). The results agree with previous work carried out by Toh et al. (2013) and Pichaiyongvongdee et al. (2014). It can be further proven that the natural active ingredient as well as phenolic compound in fruits were found in their peels and decreased in concentration when they are closer to the flesh or pulp of the fruits (Abdullah et al., 2012). Overall, the results of TPC content were significantly varied ($p < 0.05$) among pomelo residues.

Among freeze dried pomelo residues, the highest amount of TPC was exhibited in albedo (1267.87 mg GAE/100g DM), followed by flavedo (1045.14 mg mg GAE/100g DM), lamella (942.49 mg GAE/100g DM) and the lowest was pulp waste (554.65 mg GAE/100g DM). Furthermore, the influence of different drying method on TPC showed significantly varying values ($p < 0.05$) for CD and VD methods. After vacuum drying (254 and 352 mg GAE/100g DM), TPC content was reduced ($p < 0.05$) predominantly in pulp waste at 70 °C and 80 °C, respectively. It can be seen that the reduction of TPC from the extract of orange peels occurred similarly to that obtained elsewhere with hot air drying at 50 and 60 °C for 48 hr (Chen et al., 2011). In addition, relatively lower TPC value also has been reported by Sultana et al. (2012) after drying at 80 °C for different types of fruits (strawberry, mulberry, plum, apple and apricot). Conventional and vacuum drying could be used to preserve total phenolic compound as they resulted in higher retention of TPC during both post drying treatment. Basically, for conventional drying, significant variations ($p < 0.05$) of TPC was detected for lower temperature (50 – 60 °C) in comparison with higher temperature (70 - 90°C). In contrast with vacuum drying process, insignificant variation ($p > 0.05$) can be observed despite the lower and higher drying temperature, as most of the TPC showed similar retention value at any temperature. A clear trend can be observed on TPC content for different parts of pomelo residues from higher value to lower value as follows: albedo> flavedo> lamella> pulp waste. As for drying treatment, from the most trend which consist maximum value of TPC obtained to the minimum value retained in product between different drying methods is as follows: CD > VD> FD.

4.2.5 Effects of drying methods and temperature on antioxidant activity (DPPH and FRAP)

4.5.2.1 DPPH scavenging ability

Antioxidant activity affected by different drying methods of pomelo residues were evaluated using radical scavenging activity ability and reduction of Ferric compound by FRAP assay. Figure 10 presents the effects of drying methods on DPPH scavenging activity of pomelo residues at different drying temperature.

The results show that the extracted antioxidant compound possesses a good potential to neutralize the free radicals (value more than 40%) (Refer Appendix D.; Table D12). DPPH scavenging activity of pomelo residues reduced significantly ($p < 0.05$) after being dried with conventional and vacuum drying. FD was used as the control in the current study. It can be seen that freeze drying (FD) exhibited the inhibition value of DPPH at 86.21% which was significantly reduced ($p < 0.05$) by 2%, 5%,13 % and 45% after being treated during conventional drying (CD) at 60 ℃ (85 %), 70 ℃ (82%), 80 ℃ (75%), and 90 ℃ (47%) respectively. On the other hand, vacuum drying influenced DPPH inhibition activity by less than 10% at different drying temperature (50 – 90 ℃). Thus, both drying methods produced a significant effect ($p < 0.05$) on DPPH scavenging activity of flavedo.

The results showed declining trend as drying temperature increased. At drying temperature of 50 ℃, it was observed that there was improvement in DPPH scavenging activity, especially during CD. This reflects the antioxidant product generated from breakdown of the heated citrus peels still possess the antiradical scavenging of DPPH (Castro-Vazquez et al., 2016).

Similar findings support the present study for other citrus fruits which were subjected to comparable heat treatment (Jeong et al., 2004; Lobo et al., 2010; Xu et al., 2007). Nevertheless, as drying temperature applied was increased from 60 ℃ to 90 ℃, there was significant decline ($p < 0.05$) of DPPH scavenging activity during CD. This is because of longer drying time (24 hr) treatment which eventually led to a modification of chemical structure or damaging the antioxidant constituents of the fruits (Li et al., 2006). This reflects the lower productivity of available antioxidant composition to scavenge the activity of DPPH. Therefore, the results show CD at lower temperature provide better DPPH activity, while VD retain most of the antioxidant capacity at any different drying temperature (50 ℃ - 90 ℃).

In terms of pomelo residues, the FD flavedo showed the lowest value of DPPH percentage compared to other freeze-dried pomelo residues. For albedo, the value of DPPH scavenging activity in FD was 93% which was higher than the result obtained by using CD (48% - 92%) and VD (75% - 86%) at different drying temperature. It was observed that the reduction in percentage of DPPH activity was less than 50%

in conventional dried and vacuum dried albedo. The effect of both drying treatment (CD and VD) showed similar trend with flavedo part whereby the results of DPPH scavenging activity reduced consequently as drying temperature was increased. Significant variation ($p < 0.05$) of DPPH inhibition value was discovered during CD (92.25 - 48.01%) and VD (74.61 – 85.46%). This expected considering the methods used in thermal processing, the exposure of oxygen and degradation of antioxidant compound which leads to reduction in the DPPH radical scavenging activity. In addition, there was formation of new compound which have a pro-oxidant action, in other word, the pro-oxidant induce the occurrence of oxidation, rather than prevent the oxidation activities (Kong et al., 2010; Oliveira et al., 2016). The results were consistent with the result obtained by previous research which supported that FD grapefruit peel established greater scavenging activity rather than fresh and hot air-dried grapefruit peels (Castro-Vazquez et al., 2016).

A contrary trend was also observed during increased temperature for CD (90 ℃) whereby as the inhibition value of DPPH reduce (48.01%) relatively, the phenolic content (2925.63 mg GAE/100g DW) was increased (Figure 9). This is due to larger phenolic compound was breakdown into smaller compound and could not sustain extensively at higher temperature. The tendency of scavenging activity of DPPH for CD agreed with those reported by Ding et al. (2017) in lemon slices where it was observed that there were no significant changes ($p > 0.05$) of DPPH value at 50 ℃ and 60 ℃ drying temperature, while it decreased significantly ($p < 0.05$) as the temperature increased to 90 ℃. The finding is consistent with the results obtained by other researchers on lime products (Esparza-Martínez, et al., 2016).

In contrast, VD presented relatively constant value with minimal changes of radical scavenging as drying temperature. It can be concluded that the remaining antioxidant constituents in vacuum dried albedo exhibited better scavenging activity ability than after it was dried during CD at higher temperature. This mechanism is an indication of the effects of drying which led to modification of existing antioxidant composition that eventually reflects the formation of novel antioxidant and boost the initial antioxidant status. In other words, formation of low molecular weight phenolic compounds of heated citrus peels might contribute to antioxidant activities (Jeong et

al., 2004; Que et al., 2008)). Furthermore, the presence of Maillard reaction and caramelization usually occur at higher temperature and produce byproducts revealed that the methods used in preparing the samples particularly during VD. These byproducts could contribute towards antioxidative activities as well (Chen et al., 2009; Liu et al., 2007; Nicoli et al., 1999). This ability is reflected in high development of redox-active metabolites which had an important role in decomposing peroxides or adsorbing and neutralizing the free radicals as reported by previous study (Castro-Vazquez et al., 2016; Lobo et al., 2010). Similar trend was discovered by Xu et al. (2017) using vacuum dryer method to dry tangerine peels at different drying temperature (60 °C - 90 °C).

VD can preserve the antioxidant composition while maintaining the radical scavenging of DPPH for dried albedo better than CD. As for lamella, insignificant difference ($p > 0.05$) of trend with flavedo and albedo were observed by which DPPH reduced during post drying treatments. The results obtained from Figure 10 were in the range of 67% - 92% for CD whereas for VD produced around 67% -82% at different drying temperature comparing to FD (92%). There was a decrease in scavenging DPPH activities of lamella, ranging from 0.4% to 26.5%, compared to FD lamella. Furthermore, scavenging DPPH activities did not change significantly ($p > 0.05$) when the drying temperature is applied at 50 °C but increased significantly ($p < 0.05$) as the temperature further increased to 90 °C during CD. As for vacuum drying treatment, in general, the levels of DPPH antioxidant capacity for lamella reduced insignificantly at 50 °C ($p > 0.05$), while it started to increase significantly ($p < 0.05$) as the drying temperature increased to 80 °C. This could be due to the release of bound phenolic compounds based on breakdown of cellular constituents and the development of new compounds with improved DPPH value at higher temperature (Chen et al., 2009). Similar results were obtained by other authors working with tangerine peel (Xu et al., 2017) and lime (Esparza-Martínez et al., 2016).

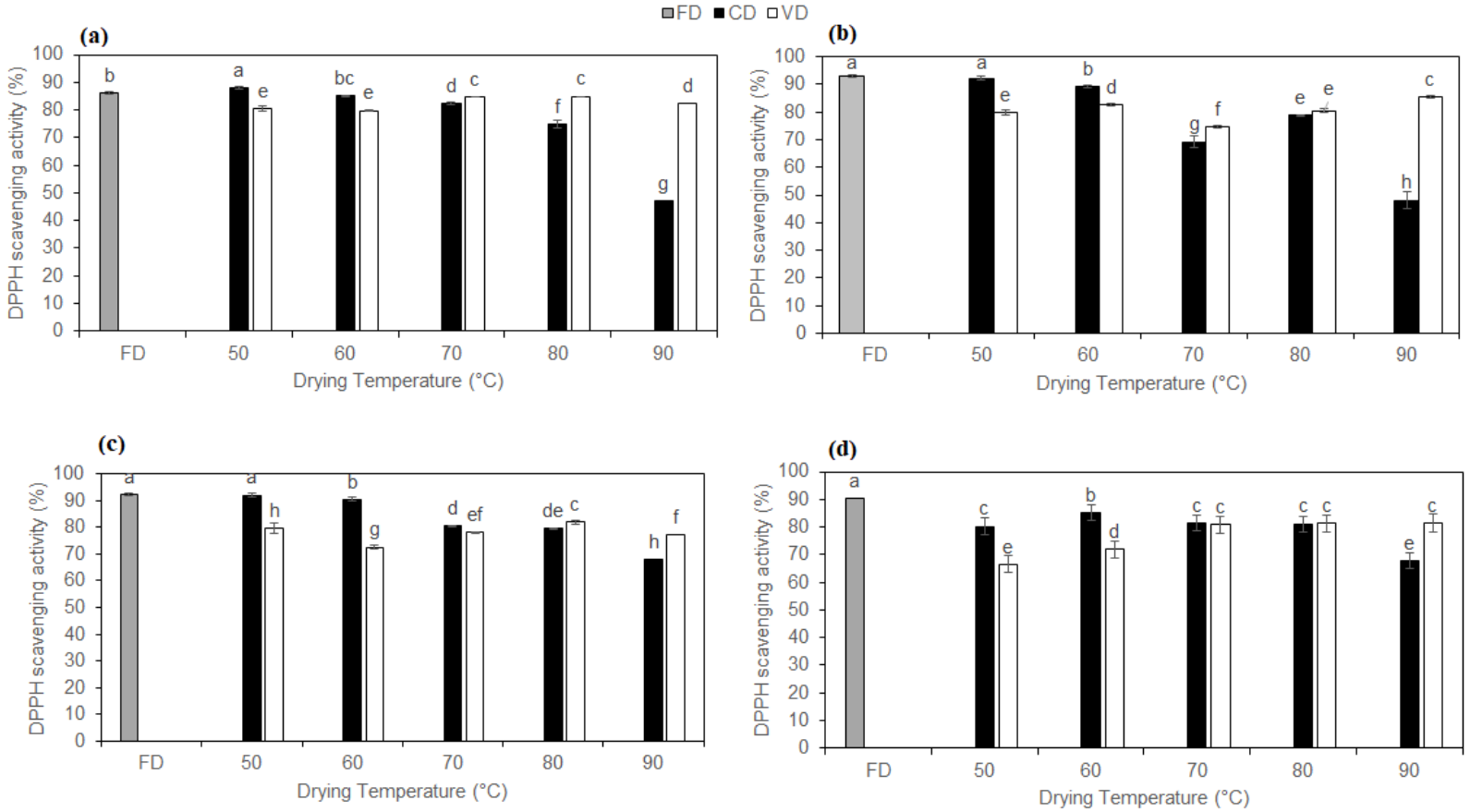

Figure 10 DPPH scavenging activity affected by freeze drying (FD), conventional drying (CD) and vacuum drying (VD) methods of pomelo residues namely (a) flavedo, (b) albedo, (c) lamella, (d) pulp waste at different drying temperature (50-90°C). The letters (a, b, c) were signifies as significant affected ($p < 0.05$) by different drying methods.

Hence, the findings suggested that CD particularly at lower temperature was a suitable drying method to maintain the DPPH activities of lamella.

As for pulp waste, the maximum % DPPH scavenging activity was observed during freeze drying (91%) followed by conventional drying (67% - 85%) and vacuum drying (67% - 82%). The potential of pulp waste being dried using both drying method with scavenging DPPH radical appearing thus showing a similar effect with other pomelo peels. The FD pulp waste showed DPPH scavenging activity to be significantly higher ($p < 0.05$) compared to other drying methods. The reason both drying methods (CD and VD) exhibiting lower DPPH scavenging activity might be due to the thermal process; particularly at lower drying temperature, lower drying rate was observed and longer time was needed to preserve the phenolic compound from the samples to be dissociated (Geankoplis, 2003). In addition, as temperature increased, certain compounds are being liberated during drying process (Jeong et al., 2004) which significantly effects ($p < 0.05$) the DPPH scavenging activity. Briefly, the findings in the current study are consistent with results obtained by Sultana et al. (2012). In addition, the decreasing trend of antioxidant activity in dried fruits might be because of the alteration in chemical compound or loss of existing antioxidant compound from the fruit byproducts (Li et al., 2006).

There is reduction of 6% during conventional drying at 60 °C, which is considered slightly significant with FD of scavenging radical activity of DPPH being a better approach in comparison with vacuum dried. The results showed significant reduction ($p < 0.05$) of more than 20% at similar temperature. Dissimilar trend was observed for both drying method with increased temperature. During CD, the trend of DPPH scavenging activity reduced significantly ($p < 0.05$) as temperature increased, and vice versa for VD methods. It could be due to major phenolic composition obtained from CD having byproducts produced after exposure to the oxygen and reacts as pro-oxidant instead of behaving as antioxidant. As claimed by Nayak et al. (2015), during the preliminary stage, reduction in antioxidative activity can be attributed not only the thermal degradation of naturally present antioxidants but also to development of early Maillard reaction products (MRPs) with pro-oxidant characteristics. Nevertheless, as vacuum drying reflects the absence of oxygen, limiting the formation of Maillard

reaction to products thus, maintaining the DPPH scavenging activity even though the major phenolic compound at different drying temperature is lesser than during CD, as shown in Figure 4.1. In other words, DPPH radical scavenging effect could be increased instead of lessened with drying time during vacuum drying (Chen et al., 2011; Ho & Lin, 2008; Jeong et al., 2004).

Generally, the overall findings illustrated a significant effect ($p < 0.05$) for DPPH value during CD and VD for pulp wastes. Similar trend was observed for flavedo and albedo. Among post drying methods, VD showed greater retention in stabilizing the inhibition of DPPH value when higher temperature was applied compared to CD, while CD established sustainable scavenging activity at lower temperature.

Overall, among the FD of pomelo residues, the antioxidant activities were superior in albedo, followed by lamella, pulp waste and flavedo. Superior antioxidant activity appeared from FD due to method used in preparing the samples whereby the samples were frozen prior to drying process which limit the degradation and retain the antioxidant compound. Furthermore, both drying (CD and VD) methods displayed significant effect ($p < 0.05$) on DPPH values in all different parts of pomelo residues. At lower drying temperature (CD), remaining antioxidant still showed positive antioxidant activity, however the effects start to decrease as drying temperature increased. There is diverse trend with VD whereby the findings showed that small effects appeared at any temperature between the ranges of 50 °C - 90 °C). Nevertheless, the dried pomelo byproducts presented a greater inhibition value of DPPH than the fresh pomelo byproducts.

4.2.5.2 Ferric reducing ability (FRAP)

Figure 11 displays the variation of drying methods on FRAP value of pomelo residues at different drying temperature. The outcomes regarding FRAP values of pomelo residues in ranges from 2.4 mM Fe (II)/g DW to 14.0 mM Fe (II)/g DW for flavedo, 1.7 to 11.4 mM Fe (II)/g DW for albedo, 1.7 to 5.78 mM Fe (II)/g DW for lamella, and lastly from 1.3 mM Fe (II)/g DW to 18.8 mM Fe (II)/g DW for pulp waste.

Flavedo, the exocarp of the pomelo fruits, the values of FRAP was significantly higher ($p < 0.05$) in conventional drying (2.6 mM Fe (II)/g DW – 14.0 mM Fe

(II)/g DW) ($p < 0.05$) compared to vacuum drying (2.4 mM Fe (II)/g DW - 4.87 mM Fe (II)/g DW) process. In addition, FD flavedo generated FRAP value 4.00 mM Fe (II)/g DW. Freeze drying flavedo established the highest FRAP value compared to lower drying temperature in CD (2.6 – 3.1 mM Fe (II)/g DW) and (VD 2.4-3.4 mM Fe (II)/g DW) at 50 °C to 70 °C respectively. This is thought to be due to the existing of phenolic content (Figure 4.1) which is capable of donating a hydrogen ion to ferric tripyridyl triazine (Fe^{3+}-TPTZ) complex and bothered the radical chain reaction (Lim & Loh, 2016). In addition, observation of oil glands (spotted dark circle) is frequently found in the stems, leaves, flowers and fruits which is located in the flavedo (exocarp)(Figure 3.4A) of the rind (Knight et al., 2001) as present in Table 4.3.

Similar findings were obtained by Chaiyana et al. (2014) which found the oil made from these oil glands possesses high antioxidant activities. Thus, the current findings present higher fat content obtained in freeze dried samples of flavedo (1.63%) in comparison to fresh (1.51%) and oven dried samples (0.73%). Nevertheless, despite the current study, the previous study by Lim and Loh (2016) reported methanolic extract of white Tambun pomelo had higher FRAP value which was 141.25 mM Fe (II)/g DW. This could be attributed to the overall peels that were collected and analyzed together which made higher FRAP values than the present study, while FRAP values signify each pomelo residue separately.

The lower FRAP values (1.08-3.63 mM Fe (II)/g FW) in current study was evaluated and compared with the FRAP values (14. 7 – 47.5 µmol Fe(II)/g FW) obtained by 21 types of citrus peel varieties (Ramful et al., 2010) which range from 1420 to 8130 mM Fe (II)/g FW). Significant increase ($p < 0.05$) of FRAP value obtained after conventional drying at 90 °C were 10 times higher than FD method. A similar tendency was reported in lemon slices where the value of FRAP at lower temperature was lower initially and increased as the temperature increased (50 °C to 90 °C) (Ding et al., 2017).

As for albedo, significant variation ($p < 0.05$) was observed after being dried in conventional dryer (2.1 – 11.3 mM Fe (II)/g DW) and vacuum oven dryer (1.8 – 4.8 mM Fe (II)/g DW) in comparison to freeze drying (2.6 mM Fe (II)/g DW) treatment. FRAP value of conventional dried albedo at 90 °C (11.33 mM Fe (II)/g DW) has been reported to be significantly higher ($p < 0.05$) than freeze dried albedo (2.6 mM Fe (II)/g

DW). The decreasing order of FRAP values from lower value to high was reported as: FD (2.5 mM Fe (II)/g DW) < VD (1.8 – 4.8 mM Fe (II)/g DW) < CD (2.1 – 11.3 mM Fe (II)/g DW). In general, highest value of FRAP signify the highest reducing power that reflects the highest antioxidant activity (Jayaprakasha, et al., 2001; Toh et al., 2013). Conventional drying produced significantly greater ($p < 0.05$) (2.05 – 11.33 mM Fe (II)/g DW) reducing power than FD (2.6 mM Fe (II)/g DW). Dried products were highly suitable to be used in grinding process and able to yield higher extracted compounds. As the extraction yield increased, it reflects the total phenolic compound concentration. Higher value of TPC content from CD albedo (1330.33 – 2925.63 mg GAE/100 g DW) is better than FD (1267. 87 mg GAE/ 100 g DW) albedo which might be due to development of new compounds from their precursor in Maillard reaction. These new products possessed potential reducing power and contribute to FRAP values (Lim & Loh, 2016; Que et al., 2008; Yu, et al., 2005; Mediani et al., 2014).

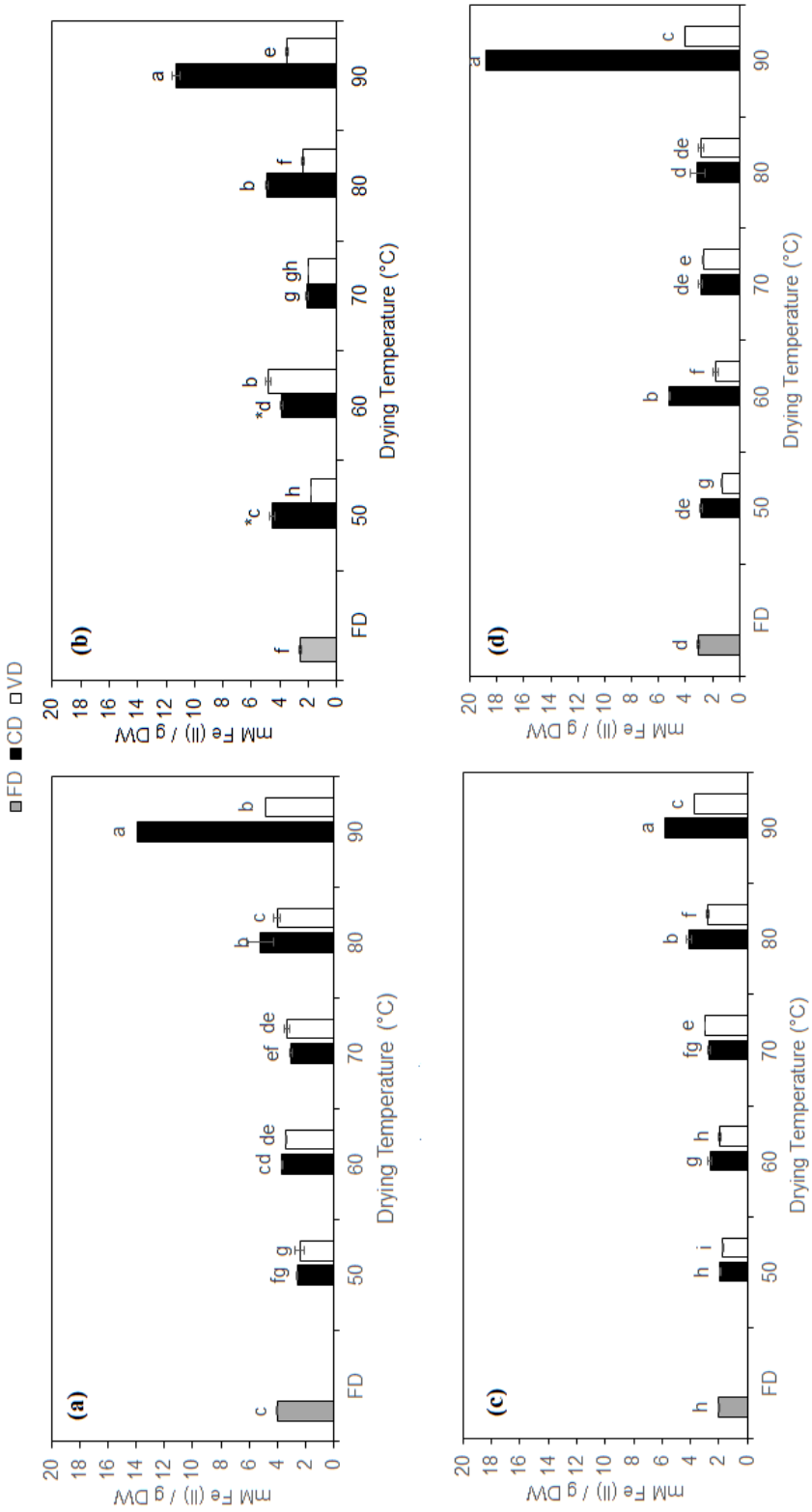

Figure 11 FRAP value affected by freeze drying (FD), conventional drying (CD) and vacuum drying (VD) methods of pomelo residues namely (a) flavedo, (b) albedo, (c) lamella, (d) pulp waste at different drying temperature (50-90°C).

In contrast, the study by Castro-Vazquez et al. (2016) indicated that the FD samples (181.10–207.74 mg Trolox/g DW) showed greater FRAP value compared to CD (105.9 and 79.4 mg Trolox/g DW) of fresh (60.3 and 44.8 mg Trolox/g DW) white and pink of grapefruit peels. The FRAP value recorded in current study was lesser than in tangerine peels at 60℃ - 90 ℃ (21.5 – 23.5 μmol /100mg dried material) during vacuum drying (Xu et al., 2017). In general, the CD albedo exhibited ($p < 0.05$) greater ability of ferric reduction compound particularly at higher temperature. It might be due to the membrane of plant cell wall containing phenolic compound were destroyed with the presence of oxygen (Uçan et al., 2016;Agcam et al., 2014). Then, higher free phenolic compound were presence during CD process which might contribute to higher ability of FRAP value.

Significant reduction ($p < 0.05$) was observed for lamella when it was conventionally dried compared to freeze drying treatment. FRAP values were reduced by 5% during CD at 50 ℃ while 13.6% during VD when compared to FD. Similar with albedo, with increased temperature during CD, significant improvement ($p < 0.05$) of reduction of ferric ability was detected. Thus, in monitoring the trend, the FRAP values of 50 ℃ – 90° C for CD generated 1.9 - 5.8 mM Fe (II)/g DW, while for VD present 1.7 – 3.7 mM Fe (II)/g DW at the same temperature in comparison to FD (2.0 mM Fe (II)/g DW).

Insignificant difference ($p > 0.05$) of FRAP value was observed between CD (50 ℃) and FD for lamella. This indicates that the high molecular weight of phenolic compound have been dissociated and simple free forms were produced when treated with heat treatment and increased as TPC content increased (Sultana et al. 2012), as confirmed in Figure 9. Ultimately the FRAP values rise correspondingly with the amount of TPC which was higher in CD lamella than FD lamella. Similar trend were evaluated with the vacuum drying process.

Lastly, for pulp waste, the FRAP values obtained from FD was 3.1 mM Fe (II)/g DW, however, it increased as CD (2.8 – 18.8 mM Fe (II)/g DW) and VD (1.3 - 4.0 mM Fe (II)/g DW) was applied during higher temperature. Similar trend of unshiu fruit peels (from 71.8 to 171.0 μM) as temperature increase (from 50 to 150 ℃) after being treated with heat treatment (Jeong et al., 2004; Keke et al., 2014). During CD at high

temperature (18.8 mM Fe (II)/g DW), the findings obtained in FRAP values was increased by 6 time more than FD (3.1 mM Fe (II)/g DW). As previously discussed, the action of a one hydroxyl group (or more) that attached with aromatic rings (i.e hesperidin) in pulp waste (Chinapongtitiwat et al., 2013) that can donate the electron could be attributed to the increase of FRAP values (Prasad et al., 2010). In addition, as pulp waste obtained by post extraction juice, the seeds of pomelo might be counted in the pulp waste remaining. Thus, the seeds were revealed as the parts of pomelo fruits which exhibited the highest antioxidant properties. As a result, higher ferric reducing power was recorded in the current study. In addition, since the major antioxidant compound in citrus contain total polyphenols, it does show and support the reason behind high potential of antioxidant characteristic (Sun et al., 2002; Pichaiyongvongdee et al., 2014).).

Generally, CD showed significant increase ($p < 0.05$) of FRAP values in pulp waste and albedo compared to lamella and flavedo and VD showed a relatively stable and minimum change of FRAP values in overall parts of pomelo residues. The conclusion about the correlation of phenolic compound reflected the TPC content which will be evaluated in Section 4.2.6. Finding the variation of antioxidant activities between pomelo residues is common as the phytochemical profiles between different species and cultivars could be varied (Balasundram et al., 2006; Kumar et al., 2018). Thus, processing at different drying condition particularly higher drying temperature (90 °C) affected the reducing ability of ferric compounds which have been liberated by heat treatment. High heat treatment could cleave the covalently bound phenolic compounds from the cell wall of residues. In addition, the results of the present study exhibited considerable variation of antioxidant activity among different parts of pomelo residues after being treated at different drying temperature.

Overall, in terms of pomelo residues, the FRAP values displayed a higher potential source as natural antioxidant in freeze dried form from flavedo followed by pulp waste, albedo and lamella respectively. In terms of drying methods, CD suggest a better method to preserve the compound and contribute to the FRAP values than VD.

4.2.6 Correlation of total phenolic content and antioxidant capacity

The relationship of TPC and antioxidant activity of pomelo residues were analyzed using Pearson's correlation (Table 30).

Table 30 Pearson correlation between total phenolic content and antioxidant activity of pomelo residues

Antioxidant Content / Antioxidant Activity	TPC
DPPH Free Radical Scavenging Assay (%)	r = - 0.475**
Ferric Ion Reducing Antioxidant Power Assay (FRAP Value)	r = 0.413**

r = Pearson coefficient

**Correlation were significant at the 0.01 level (2 tailed)

Moderate negative correlation between TPC and DPPH values have been recorded in the present study. Clear negative correlation was discovered between TPC of the pomelo residues and DPPH free radical scavenging activity (r = - 0.475), which signifies that the inhibition of DPPH free radical was not directly influenced by the existence of phenolic content. The relationship was statistically significant ($p < 0.05$) between TPC and radical scavenging activity of DPPH. Therefore, the phenolic contents (TPC) are not superior bioactive compound which can directly reflects DPPH scavenging activity, because some of phenolic compound might not have good free radical scavenging ability. Generally, in most cases, pomelo residues have greater phenolic content and exhibited lower DPPH values. On the other hand, naturally antioxidant compound including ascorbic acid, limonoid, fiber and dietary fiber in pomelo peel may correspond and act as anti – oxidant as well (Breksa & Manners, 2006; Mat Zain et al., 2014; Pichaiyongvongdee & Haruenkit, 2009). The findings were supported by Lim and Loh (2016) which reported a significant relationship ($p < 0.05$) between TPC and DPPH free radical scavenging analysis (r = - 0.853). However, contrary findings were reported by previous research whereby, a positive correlation were observed between TPC and DPPH in citrus fruits (Abd Ghafar et al., 2010; Chang & Azrina, 2017; Jang et al.,

2010; Pichaiyongvongdee et al., 2014). The difference might be due to the difference in variety of citrus used and method of sample preparation employed.

Antioxidant activity might not usually relate well with presence of phenolic content (Ghasemi et al., 2010). This is because certain phenolic compound might not possess a good free radical scavenging ability (Jang et al., 2010). Previous research showed that flavonoid compound with particular structure with hydroxyl groups attached could be a potential source of proton donor and generate anti- radical scavenging activity (Chang & Azrina, 2017; Hou et al., 2003; Mensor et al., 2001).

A positive relationship was observed between TPC and FRAP, which reflects on the ability of the phenolic compounds while chlorogenic acid and naringin (28.552 g NAR/ kg dry based) attributed to the TPC which existed in seeds among the pulp that still remained in pulp waste during post juice extraction. The presence of naringin and limonin as reported by was spread in varying amounts around pomelo fruits which may correspond to the FRAP values (Pichaiyongvongdee and Haruenkit, 2009; Pichaiyongvongdee et al., 2014).

In the present study, a moderate relationship (r =0.413) was recorded between TPC and FRAP values, where significant ($p < 0.05$) coefficient value were obtained. Thus, it can be assumed that, FRAP analysis related very well with other antioxidants although antioxidant activities might not commonly possess strong relationship with phenolic compound (Toh et al., 2013). The presence of ascorbic acid might react and corresponds well with the ferric reducing ability (Abeysinghe et al., 2007; Chang & Azrina, 2017; Guo et al., 2003; Toh et al., 2013). However, the correlation between FRAP value in fruits was varied.

4.3 Selection of pomelo residues and best drying condition

4.3.1 Selection of pomelo residues

Phenolic compounds are present in high amounts of citrus peels and exhibit nutraceutical which are beneficial mainly because of their antioxidant activities. The presence of phenolic compound was assessed by total phenolic compound (TPC) values as briefly explained in Section 3.7.2. Comparison of total phenolic content

between different pomelo residues are shown in Figure 12. The phenolic compounds of citrus peels are considerably different according to the parts of pomelo peels.

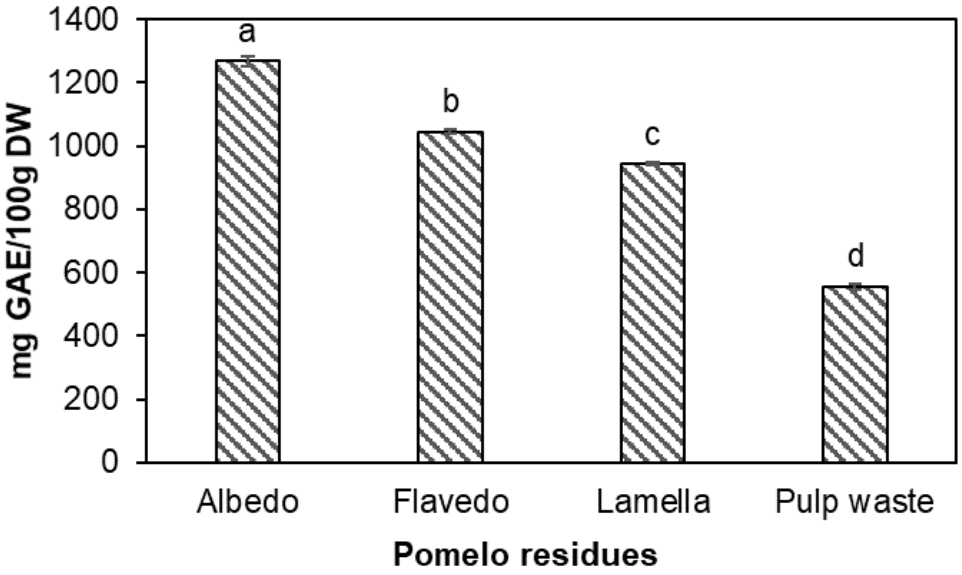

Figure 12 Comparison of total phenolic content of pomelo residues

In the current study, albedo was selected with the most amount of total phenolic content compared to other parts of pomelo residues. Thus, selection of best drying condition of albedo was carried out in the current study at Section 4.3.2.

4.3.2 Selection of ideal drying condition

Table 31 presents the minimum values of moisture content (MC), maximum total phenolic content (TPC) and percentage inhibition of DPPH (%) of albedo.

Table 31 Factorial design for two factors and results of MC, TPC and DPPH of pomelo albedo

Drying methods	Temperature (°C)	MC (g/100g DM)	Inhibition of DPPH (%)	TPC (mg GAE/100g DW)
CD	60	5.88	89.26	1336.77
VD	60	9.72	82.70	958.30
CD	70	3.32	69.24	2007.46
VD	70	8.95	74.61	2099.18
CD	80	3.72	78.71	2370.10
VD	80	7.58	80.49	2608.42
CD	90	4.64	48.01	2925.63
VD	**90**	**8.57**	**85.46**	**2516.51**
CD	50	6.53	92.25	1330.33
VD	50	20.26	79.75	1790.94
Control		8.63	93.03	1267.87

CD: conventional drying; VD: vacuum drying; MC: moisture content; TPC: total phenolic content; DPPH: 2, 2 – Diphenyl-1-picrylhydrazyl

The impacts of drying methods and temperature on MC albedo can be reffered to Section 4.2.2.2, on TPC in Section 4.2.4 and DPPH scaveging activity in Section 4.2.5.1. The effects of drying methods and temperature on MC, TPC and DPPH of albedo were summarized in Appendix G. The predicted MC, TPC, DPPH were 7.86%, 2727.19mg GAE/ 100DW and 82.44% respectively. These predicted values are closer to their corresponding experimental values of 8.57%, 2516.51mg GAE/100g DW and 85.46%. The overall desirability of 0.800 were obtained based on albedo with the target of minimum value of MC (less than 10%), maximum value of TPC and DPPH (Figure 13) The results obtained for the vacuum drying methods, the kinetics, and the

quality of dried pomelo albedo. The interconnection and its significance between the parameter can be refer to Appendix G.

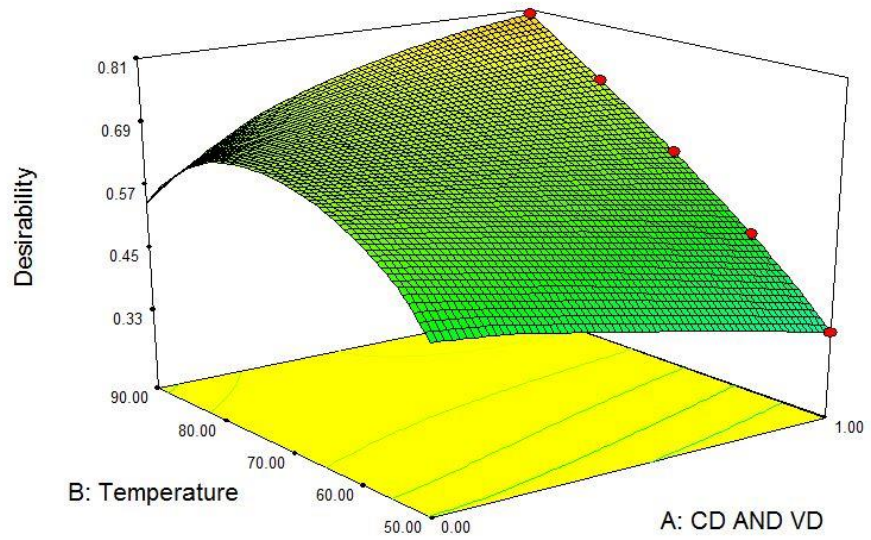

Figure 13 Effects of drying methods and drying temperature on 3D plot of the desirability index for the pomelo albedo

In the range of the factors used, 95% confidence prediction gave optimal vacuum drying process at drying temperature 90 °C. These conditions were used to identify the effects of storage time on selected pomelo residues crude extract (albedo) and kinetic modeling reaction during storage period can be refer to Section 4.5.

4.4 Drying kinetics at different drying temperature between conventional and vacuum drying condition

4.4.1 Drying kinetics

Data from the moisture ratio versus time are presented in Figure 14(a, b) for conventional drying and vacuum drying respectively**.** The pomelo albedo was dried as a single layer with the thickness approximately ~10 mm at the drying temperature of 50, 60, 70, 80 and 90 °C using conventional and vacuum dryer. The deviation in moisture ratio of pomelo albedo as a function of drying time at different temperature for conventional drying and vacuum drying (VD) are shown in Figure 14. The results show moisture ratio of albedo in both corresponding drying methods reduced

significantly ($p < 0.05$) with increase in drying temperature. Samples for albedo with an initial moisture content of 77.69 kg water/kg dry matter reduced to a final moisture content of less than 10% in value. It indicates that increasing the drying temperature decreases the drying time significantly ($p < 0.05$). Heat transfer rate between the material and thermal source increased eventually leading to the generation of a larger driving force resulting in rapid evaporation of water and shorter drying time at high drying temperatures (Onwude et al., 2016; Wang et al., 2018)Torki-Harchegani et al., 2016; Xu et al., 2017).

During VD process (Figure 14b), moisture takes a longer time to evaporate than CD, therefore, drying time of VD was longer. In a vacuum oven, the moisture on the surface of samples evaporates quickly, however, moisture of the materials in the samples could not migrate to surface in time due to the existence of film on the surface of the materials which retarded the internal moisture migration and eventually prolonged the drying time (Xu et al., 2017).

In addition, during initial drying period under conventional drying (CD), reflected that free moisture migrated from the surface of the product to the hot air environment which is known as first falling rate period. In the second falling rate period, the distribution of external heat diffusion into the inner cell of pomelo albedo becomes slower, due to the hardened surface (Refer to Appendix E, Figure E2) of pomelo albedo. This condition allows limited moisture transfer from the centre of pomelo albedo to evaporate and diffuse towards the surface and to the hot air boundary; therefore, drying rate becomes slower as drying time increases (Varith et al., 2007). Consistent with another citrus, drying lemon fruits also falls into a category of falling rate period which concludes the diffusion mechanism mainly contributes to the moisture movement (Darvishi et al., 2014; Torki-Harchegani et al., 2016). The result is in line with earlier literature that found drying temperature effect on the the drying duration of grapefruit seed (Cantu-Lozano et al., 2013), dried lime fibre (Jongaroontaprangsee et al., 2014), lemon slices (Torki-Harchegani et al., 2016) and tangerine peels (M. Xu et al., 2017).

The moisture ratio was calculated using Equation 3.16 at specific condition and they were fitted to seven selected thin layer drying models listed in Table 26 which

has been applied using citrus based products (Torki-Harchegani et al., 2016). These models were evaluated based on coefficient of determination (R^2), standard error estimate (SEE) and root mean square error (RMSE) (Adiletta et al., 2016; Onwude et al., 2016; Torki-Harchegani et al., 2016). The results obtained showed goodness of fit parameters at different drying temperatures (50 - 90°C) for conventional drying and vacuum drying (VD) in the proposed models as shown in Table 32.

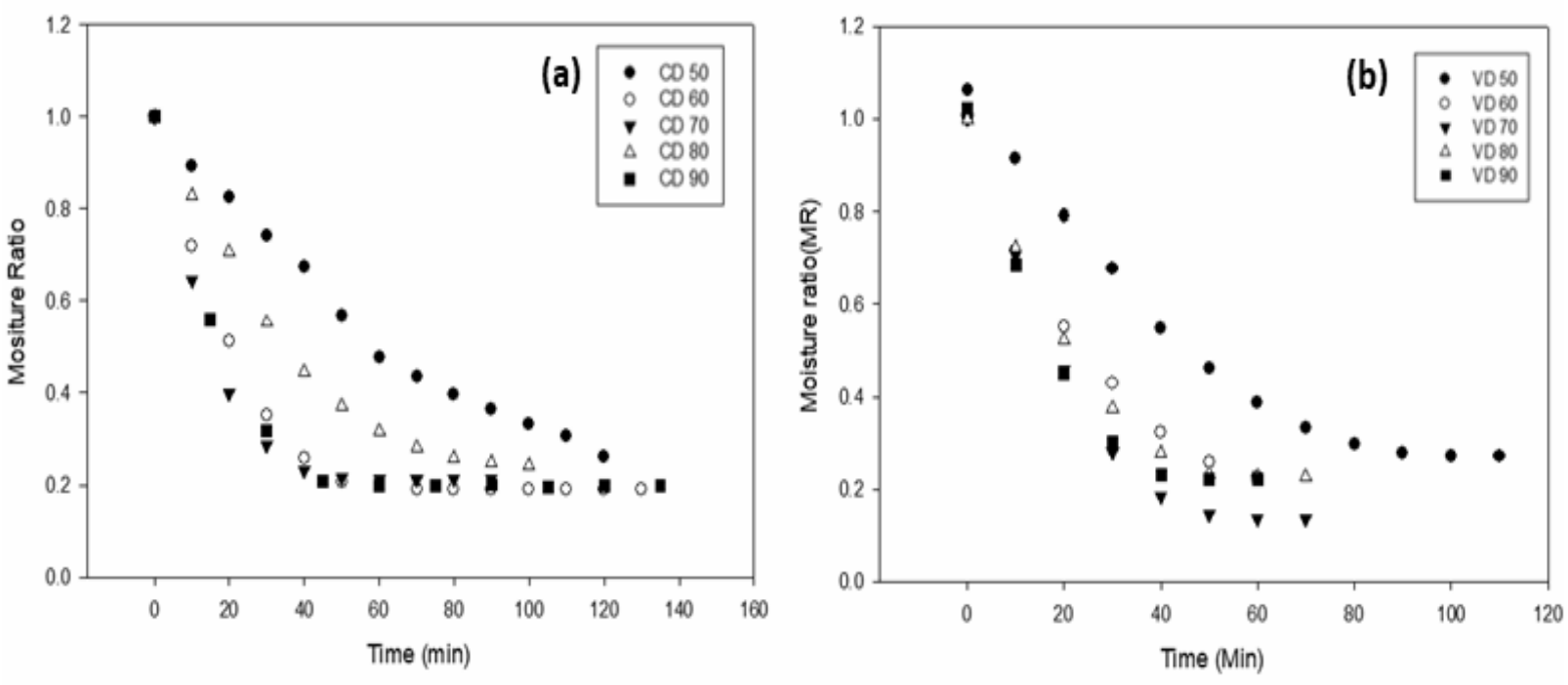

Figure 14 Drying kinetic of dried albedo during a) conventional and b) vacuum drying at 50, 60, 70, 80 and 90 °C.

Table 32 Goodness of fit parameters for selected mathematical models under conventional drying and vacuum drying method.

Model name	Temperature (°C)	Drying methods					
		Conventional drying (CD)			Vacuum Drying (VD)		
		R^2	SEE	RMSE	R^2	SEE	RMSE
Newton	50	0.9938	0.0192	0.0184	0.9793	0.0397	0.038
	60	0.8437	0.0985	0.0949	0.9923	0.0246	0.0228
	70	0.8522	0.1011	0.0959	0.9874	0.036	0.0337
	80	0.9801	0.037	0.0353	0.9698	0.0492	0.0461
	90	0.7979	0.1182	0.1121	0.9682	0.0544	0.0504
Henderson and Pabis	50	0.9943	0.0193	0.0177	0.9861	0.034	0.0311
	60	0.8612	0.0966	0.0894	0.9939	0.024	0.0203
	70	0.8707	0.1003	0.0897	0.9874	0.0389	0.0337
	80	0.981	0.0381	0.0344	0.9723	0.0509	0.0441
	90	0.8172	0.1192	0.1066	0.9683	0.0595	0.0503

Table 32 *(Continued)*

Model name	Temperature (°C)	R^2	SEE	RMSE	R^2	SEE	RMSE
Wang and Singh	50	0.9956	0.017	0.0156	0.9887	0.0308	0.0281
	60	0.8809	0.0895	0.0828	0.9946	0.0225	0.019
	70	0.9121	0.0827	0.0739	0.9959	0.0221	0.0191
	80	0.9981	0.0119	0.0108	0.9966	0.0178	0.0154
	90	0.8249	0.1167	0.1043	0.9954	0.0226	0.0192
Logarithmic	50	0.9943	0.0202	0.0177	0.9932	0.0252	0.0218
	60	0.9928	0.023	0.0204	0.999	0.0106	0.008
	70	0.996	0.0188	0.0157	0.9927	0.0323	0.0256
	80	0.9946	0.0216	0.0184	0.9953	0.0228	0.0181
	90	0.995	0.0212	0.0177	0.9953	0.0256	0.0193
Midilli and Kucuk	50	-2.0252	0.4897	0.4074	-1.206	0.48	0.3919
	60	-0.0505	0.2911	0.246	-0.439	0.4753	0.3111
	70	0.132	0.3	0.2324	0.1024	0.4017	0.2841
	80	-0.9537	0.4377	0.2527	-0.247	0.4183	0.2957
	90	0.3985	0.2496	0.1934	0.0386	0.4231	0.277

Table 32 *(Continued)*

Model name	Temperature (°C)	R^2	SEE	RMSE	R^2	SEE	RMSE
Diffusion approach	50	**0.995**	**0.019**	**0.0166**	0.9818	0.0411	0.0356
	60	**0.9922**	**0.0239**	**0.0212**	0.9991	0.0101	0.0076
	70	**0.9958**	**0.0193**	**0.0161**	0.9922	0.0336	0.0266
	80	**0.9939**	**0.023**	**0.0196**	0.9952	0.0232	0.0184
	90	**0.9948**	**0.0214**	**0.0179**	0.9931	0.0311	0.0235
Two term exponentials	50	0.9943	0.0213	0.0177	**0.9932**	**0.0267**	**0.0218**
	60	0.9928	0.0241	0.0204	**0.9991**	**0.0117**	**0.0076**
	70	0.996	0.0203	0.0157	**0.9927**	**0.0361**	**0.0256**
	80	0.9946	0.0231	0.0184	**0.9953**	**0.0255**	**0.0181**
	90	0.995	0.0229	0.0177	**0.9953**	**0.0295**	**0.0193**

These statistical tests evaluated the goodness of fit on the experimental data and they have been used in various food drying studies (Nesrine et al., 2015). The individual constant parameters of the best drying models between conventional drying and vacuum drying (VD) are shown in Table 33.

Table 33 Parameters of drying models between conventional drying (CD) and vacuum drying (VD)

Model name	Temperature (°C)	Parameters	Drying method	
			Conventional drying (CD) - Diffusion approach	Vacuum Drying (VD) -Two term exponential
Diffusion approach	50	a	-0.0361	0.9441
		k_1	0.0991	0.0209
		b	0.1182	0.1407
		k_2	-	5.95×10^{-18}
	60	a	0.8261	0.428
		k_1	0.0491	0.0559
		b	1.22×10^{-17}	0.5707
		k_2	-	0.0173
	70	a	0.8031	0.9507
		k_1	0.0665	0.0465
		b	9.84×10^{-18}	0.0686
		k_2	-	1.97×10^{-17}
	80	a	0.8514	0.8446
		k_1	0.0251	0.045
		b	7.08×10^{-17}	0.1644
		k_2	-	6.84×10^{-18}
	90	a	0.8136	0.8668
		k_1	0.058	0.0568
		b	8.73×10^{-18}	0.1672
		k_2	-	5.40×10^{-18}

For conventional drying method, the values of R^2 of the diffusion approach model was more than $R^2 = 0.99$ indicating good fit for all drying temperatures and a minimum value of SEE < 0.24 and RMSE < 0.021 for selected model is indicated. For vacuum drying method, the values of R^2 of two term exponential method also found more than $R^2 = 0.99$ indicating a good fit compared to other models, and also a minimum value between the range of SEE (0.0117 - 0.0361) and RMSE (0.076 - 0.0295) for 50,60,70,80 and 90°C. Thus, this model was selected and considered as a suitable tool to predict the drying process for thin layer CD and VD process for drying pomelo albedo.

This study indicates that the drying kinetics increased as higher temperature treated in both drying methods while retaining the phenolic content of the final product. Close fit of both models including Diffusion approach for conventional drying and two-term exponential approaches for vacuum drying can be used as practical references for the industrial sector to preserve the bioactive compound in pomelo residue during processing. Highly positive antioxidant capacities retained during both thermal dryings comparable with freeze-drying. Hence, this technology lessens the drying time and consequently reduce the energy costs in comparison with the conventional process.

4. 4. 2 Correlation between experimental and predicted model of conventional drying and vacuum drying of pomelo albedo

The correlation between the experimental and predicted moisture ratio (MR) at different drying conditions are shown in Figure 15 based on diffusion approach and two term exponential model during CD and VD, respectively. The correlations between the coefficients of related drying model and oven temperature were significant ($p < 0.05$) with $R^2 > 0.99$ and SEE value less than 0.1. The proposed model provided conformity between the experimental and predicted MR. There is a_very good agreement between experimental and predicted MR values which closely band around 45° straight line.

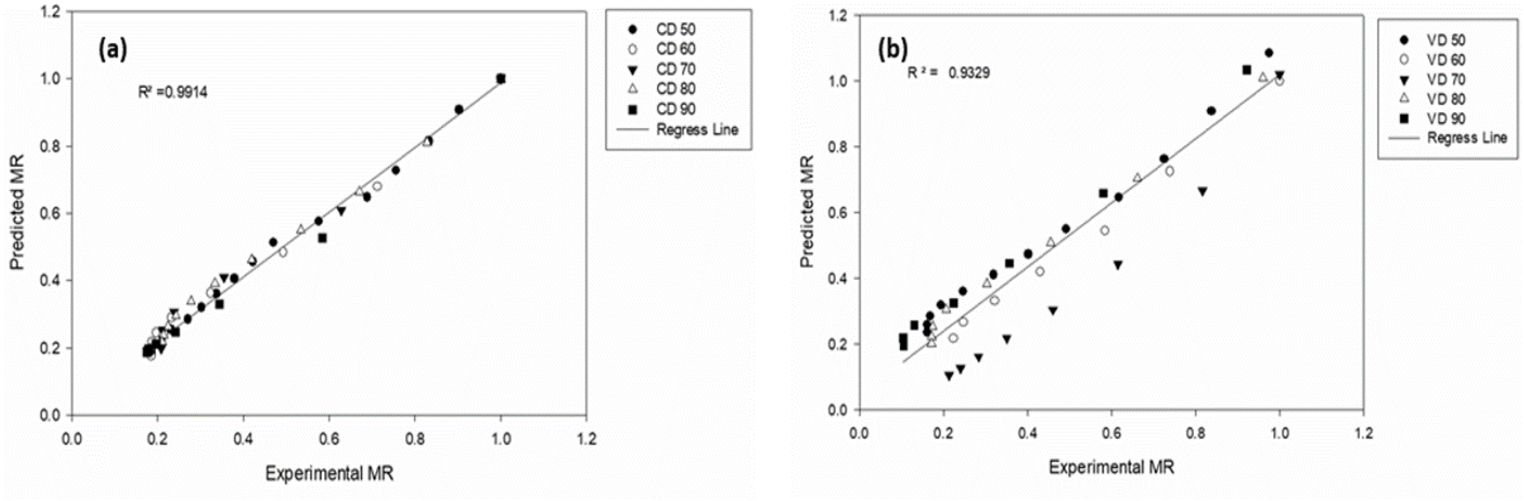

Figure 15 Correlation between experimental and predicted model based on selected models of conventional drying a**)** Diffusion approach model and vacuum drying b**)** Two-term exponential model of drying pomelo albedo**.**

In addition, based on the regression analysis, the selected model parameters for both drying methods are shown in Table 34 as functions of drying temperature.

Table 34 Relationship between drying parameter of selected model and temperature of pomelo albedo

Drying method	Selected modelling	Parameter	Equation	R^2
CD	Diffusion approach	a	$11.04 + \frac{(-2294)}{T} + \frac{169800}{T^2} + \frac{(-4138000)}{T^3}$	0.992
		b	$\frac{(-1.515 + 338.2)}{T} + \frac{(-24870)}{T^2} + \frac{601900}{T^3}$	0.998
		k	$\frac{(0.04362 + 0.000484)T}{1} + \frac{(-0.0399)}{T} + 0.00032T^2$	0.745
VD	Two Term exponential	k_1	$1.42 + \frac{(-283)}{T} + \frac{19160}{T^2} + \frac{(-425300)}{T^3}$	0.999
		k_2	$7.80 \times 10^{-17} + \frac{(-8.30 \times 10^{-19})T}{1} + (-0.01667)T$	0.999
		b	$0.1453 + \frac{(-0.002332)T}{1} + (-0.01651)T$	0.967
		a	$1 + \frac{(-0.01671)T}{1.102} + (-0.01847)T$	0.957

By applying the obtained equation in the selected model for CD and VD respectively, within the experimental conditions, the moisture content of the pomelo albedo at any time during the drying process could be calculated. This regression analysis was also used in a study by Torki-Harchegani et al. (2016) which reported Midilli and Kucuk model was a suitable model for drying lemon slices and included the parameter's models as a function of drying temperature.

4.4.3 Determination of effective diffusivity (D_{eff}) coefficients

The effective moisture diffusivity values are in the range of 5.00 x 10^{-7} ~ 6.98 x 10^{-7} m^2/s for the samples dried at 60, 70, 80 and 90 °C for CD, meanwhile for VD the diffusivity values are in the range of 9.86 x 10^{-7} ~ 1.30 x 10^{-6} m^2/s as presented in Table 35.

Table 35 Values of effective diffusivities and activation energy obtained from dried pomelo albedo at different drying temperatures for CD and VD

Drying methods	Temperature (°C)	Effective diffusivity (D_{eff}) (m^2/s)	D_o (m^2/s)	Energy activation (kJ/mol)	R^2
CD	60	5.00×10^{-7} d	4.83	9.20	0.9933
	70	6.92×10^{-7} c			0.6682
	80	6.23×10^{-7} cd			0.6993
	90	6.98×10^{-7} c			0.8971
VD	60	9.86×10^{-7} b	3.10	19.29	0.9365
	70	1.21×10^{-6} a			0.9588
	80	1.22×10^{-6} a			0.9589
	90	1.30×10^{-6} a			0.9158

Values are given as means of three replicates. Means followed by the same superscript in the same column are not significantly different at $p>0.05$ based on Duncan's Multiple range test. CD: conventional drying, VD :Vacuum drying

A higher D_{eff} rate of VD was observed in comparison with CD. The values of D_{eff} obtained from this study are within the general range 10^{-11} – 10^{-6} m^2 s^{-1} for drying of food materials and this is comparable with other values (Olanipekun et al., 2015; Zogzas et al., 1996). In addition, it was noted that D_{eff} increased constantly with the increase of drying air temperature for both corresponding CD and VD methods. This might be explained by the increased heating energy from the drying operation, which could increase the activity of water molecules and reduce the water viscosity subsequently resistance of fluid out-flow thus facilitating the moisture diffusivity in the product capillaries (Torki-Harchegani et al., 2016; Yan et al., 2010). These findings agree well with lemon slices drying using hot air drying temperature at 50, 60 and 75 °C with obtained values of D_{eff} (1.62×10^{-11}, 3.25×10^{-11} and 8.11×10^{-11} m^2 s^{-1} respectively) (Torki-Harchegani et al., 2016). For lemon slices using far-infrared radiation heating assisted pulsed vacuum dryer at drying temperature (60, 65, 70, 75°C) obtained min to maximum level of D_{eff} (1.66×10^{-11} and 1.90×10^{-10} at 60 and 75 °C), respectively (Wang et al., 2018), and for grapefruit seeds drying at temperature of 40, 50, 60 and

70°C results obtained were within range value of 4.36×10^{-10} to 6.82×10^{-10} m^2 s^{-1} (Cantu-Lozano et al., 2013). Overall, result in moisture diffusion value for each material differs for each type of materials as it depends mostly on the type of drying process involved, initial and final moisture content of the product and drying condition involved (Torki-Harchegani et al., 2016).

From Table 35, it can be seen that D_{eff} values for the vacuum dried samples are greater than those obtained for the conventional drying (CD) method. Yılmaz et al. (2017) resulted in shorter drying times with higher drying rate and higher effective diffusion coefficient in vacuum drying than conventional drying at 40~70°C for pomegranate fruit leather.

4.4.4 Determination of activation energy (E_a)

The effects of drying air temperature on the effective diffusivity are generally explained using Arrhenius' equation to generate a comprehensive agreement of the experimental and predicted data (Alara et al., 2017). The plot Ln D_{eff} versus 1/T as shown in Figure 16 shows a linear regression. The equation of effective diffusivity is shown in equation 3.23, where the activation energy (E_a) value was obtained from the slope and intercept at 9.20 kJ/mol for CD and for VD it was 19.29 kJ/mol. The value obtained for activation energy in this study is in agreement within the range of 1.27-110kJ/mol for various food (Aghbashlo et al., 2008; Torki-Harchegani et al., 2016). Lower E_a value of this process due to the high surface area of albedo exposed to hot air by convection increases the rate of moisture evaporate that require lesser energy (Xu et al., 2017) during CD drying process in comparison with VD methods.

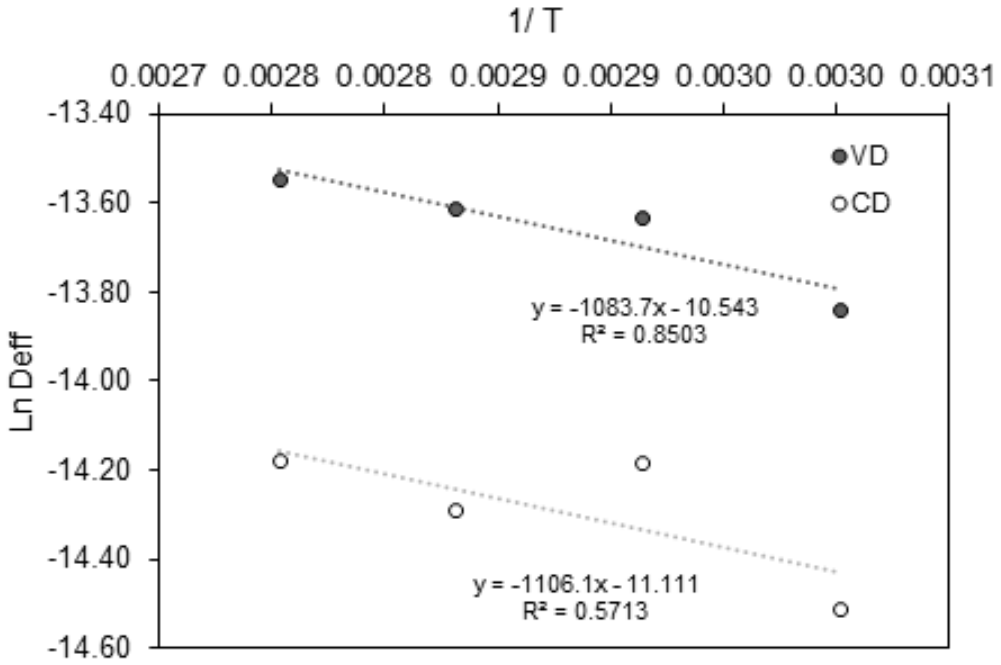

Figure 16 Relationship between effective diffusivity and temperature based on Arrhenius' model of dried pomelo albedo

The activation energy of the present study is lower than the previous study done for dried lemon (60 kJ/mol) using hot air drying at drying temperature 50,60 and 70°C (Torki Harchegani et al., 2016), grapefruit seeds (11.29 ~ 12.23 kJ/mol) at 40, 50, 60, and 70°C (Cantu-Lozano et al., 2013). Meanwhile for pulsed vacuum drying of wolfberry showed 40.08 kJ/mol when drying at (50~65°C) (Xie et al., 2018) and two different heating ways carried out by Xie et al. (2017) of far-infrared radiation (PVD-FIR) and electronic panel contact (PVD-EPC) for heating temperatures (60, 65, and 70°C).

4.4.5 Correlation of the drying parameter with effective diffusivity

A Pearson's correlation was analyzed to determine the relationship between the drying parameter and effective diffusivity (D_{eff}) of pomelo albedo (Table 36).

Table 36 Pearson correlation between drying parameter of CD and VD and D_{eff} of dried pomelo albedo

Drying method	Selected drying model	Selected parameter	Effective Diffusivity D_{eff}
CD	Diffusion approach	a	r = 0.642**
		k	r =0.089
		b	r = -0.707**
VD	Two-term exponential	a	r = 0.285
		k_1	r = 0.872**
		b	r = -0.305
		k_2	r = -0.404

r = Pearson coefficient

[a] Correlation were significant at the 0.01 level (2-tailed)

There was a very strong positive correlation between parameter (a) in diffusion approach model equation with D_{eff} compared to other parameters for conventional drying (CD) method. It was clearly shown that the selected parameter (a) of CD showed positively correlated (r = 0.642) with the value of D_{eff} which implied that higher value of parameter 'a' eventually reflects the drying kinetic of pomelo albedo and reduce the drying rate mechanism. A similar trend was observed with the two- term exponential model equation for vacuum drying (VD) method where there is a strong correlation (r = 0.872) between the selected parameter (k_1) and D_{eff}, in comparison with other drying parameters.

In brief, different parameters from selected drying model should be carried out to control specific parameter during the drying process to obtain the suitable/reduce

the drying time and achieve the target moisture content of dried albedo. By using this consideration, the result obtained could save much more time without consideration of other parameters involved to explain the complex drying process. In addition, with this approach, the experiment which usually take a long time to monitor can be avoided.

4.5 Storage studies of crude extract pomelo albedo

Drying process is considered as preliminary and significant phase in pomelo residues processing as the process can affect the composition of the final product significantly. Consequently, with moisture content being reduced, shelf life would improve, reducing storage and transportation cost in pomelo peel processing while facilitating the handling in the process. Drying may affect the existing structure of bioactive compounds such as phenol compositions or vitamin C in the materials and also correspond to alteration of original structure which causes irreversible modification to the cell wall polysaccharides. Thus, targeted quality or parameter of the dried by-products (crude) is necessary to be investigated whereby the rate of degradation of the compounds could occur during storage analysis. In addition, identification of degradation kinetic parameters related to loss of food quality is essential for predicting shelf life. Kinetic parameters, such as reaction rate constant (k), half-life ($t_{1/2}$), temperature coefficient (Q_{10}) and activation energy (E_a) are often used to estimate the shelf life of nutritional quality, and in developing storage systems that retain phenolic compounds and antioxidant activity in fruits and vegetable products (Kim et al., 2018; Ling et al., 2015). This section presents the results and discussion using several kinetic models by comparing the highest R^2 values and lower value sum of error estimation (SEE). The storage was carried out based on selected drying process (VD) at 90 °C and control (FD) which have been pre-treated prior to extraction of naringin. CD is not included in the storage of current study due to the aims of the effects of storage time on naringin being pre-treated using optimum drying condition (VD90) with control group (FD).

4.5.1 Extraction yield of phenolic compounds of pomelo albedo

Drying method (freeze drying) affected the extraction yield significantly ($p < 0.05$) and yield per cent obtained was higher in vacuum drying process (Table 37). It indicates that drying impart a positive effect on the extraction of phenolic compounds.

Table 37 Extraction yield and naringin content of freeze-dried and vacuum-dried of pomelo albedo's extract

Drying treatment	Yield of extract (g crude extract/100g db)	Naringin content (g NAR/kg db)
FD	45.06 ± 1.59[a]	16.53 ± 0.96 [b]
VD	41.97 ± 0.56[b]	28.52 ± 0.44 [a]

The difference of letters (a,b) shows significantly ($p < 0.05$) different between different drying treatments.

Extraction yield was highest in freeze dried samples (45.06g/100g DW) followed by vacuum dried (41.97/100g DW). The results obtained in this study are comparable to the yield percentage of lime (15.80g/100g) but lower than the orange variety (37.27g/100g) grapefruit (50.13g/100g) and lemon (44.68g/100g) evaluated by Guimares et al. (2010).

4.5.2 Effect of storage time on TPC of pomelo albedo

The current section focuses on the effects of storage time on total phenolic content of crude extract pomelo albedo as shown in Figure 17.

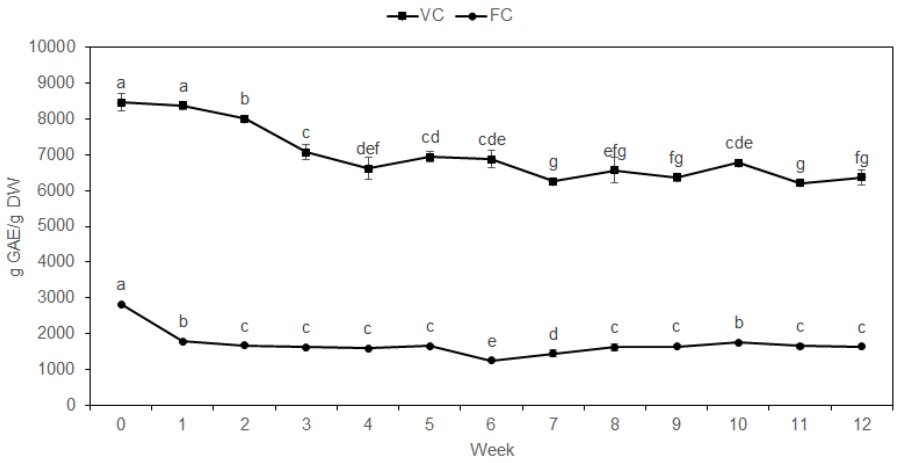

Figure 17 The effect of storage time on TPC of pomelo albedo's extract

The pomelo crude extracts were indicated as freeze dried crude extract (FC) and vacuum dried crude extract (VC) using freeze drying and vacuum oven drying, respectively. Both crude extracts have been analyzed during 12 weeks at 8 °C. Subsequently, the FC showed significant loss ($p < 0.05$) in TPC with increased storage time. The data presented 2,815 g GAE/g DW of phenolic content in day 0 for FC. On the first week, TPC content was significantly reduced ($p < 0.05$) to 1,783.9 g GAE/g DW with the differences in percentage of 36.63%. A minor fraction of phenolic content was reduced by 6% after 1st week of storage. In comparison to week 2, TPC loss was increased up to 7%. The increasing loss during food processing are sample preparation, handling and transportation, as these procedures could capture/entrap oxygen and increase degradation at different rates (Uckiah et al., 2009). In contrast with past study, loss of TPC in grapefruit juice of refrigerated and frozen juices ranged between 15 to 20% respectively (Igual, et al., 2011) which due to high moisture content and attracts microbes to actively spoiled the juices.

The results of VC show the range of TPC during the 3 month storage are between 8,454.5 and 6,209.0 g GAE/g DW. Nevertheless, VC exhibited higher TPC by 66% more than FC. In general, the application of thermal treatment (VC and FC) caused a significant ($p < 0.05$) reduction in the content of phenolic content. Phenolic compound of freeze dried and vacuum dried crude extracts showed significant differences ($p < 0.05$) during storage period. In general, the phenolic content

significantly diminished ($p < 0.05$) during 7 weeks of storage. The reduced level of TPC in dried crude extract might be attributed to chemical, enzymatic or thermal decomposition of phenolic compounds and flavonoids (Rafiq et al., 2019).

At week 9, the content of TPC showed a slight increase up to week 10, after that VC reduce significantly ($p < 0.05$) until week 12. During initial to first week, TPC of VC (from 8454.5 to 8373.3 g GAE/g DW respectively) showed insignificant results ($p > 0.05$) reflect to stable compound compared to FC (from 2815.1 to 1783.9 g GAE/g DW respectively). This can be explained by their stable radical intermediates, which prevent the oxidation of various food ingredients. Meanwhile, the loss in FC was greater than in VC stored samples. VC treatment best preserved the TPC during storage due to higher value of retention that was 3 times higher than FC. This is likely to related to attribution of high drying temperature applied which could cause the degradation of lignin and other polysaccharides complexes leading to a further phenolic acid release (Yan and Kerr, 2013; Issis et al., 2019).

The phenolic compound was retained from the 1st week until the 4th week and stabilised for 3 weeks in a row, while showing significant reduction ($p < 0.05$) during the 7th week of storage. This is due to the decomposition of chemical compounds with antioxidant properties during the storage period (Tummanichanont et al., 2018). Similarly, with study done by Wang et al. (2000), reduction of phenolic content was observed during storage. The mechanism described the reduction of phenolic was due to oxidation or polymerization during processing and storage. Nevertheless, after 10th week storage till 12th week, insignificant increase ($p >0.05$) of about 6% was observed.

4.5.3 Effect of storage time on naringin

Storage analysis was done to identify the stability of naringin while being stored under 7 – 8 °C (Figure 18).

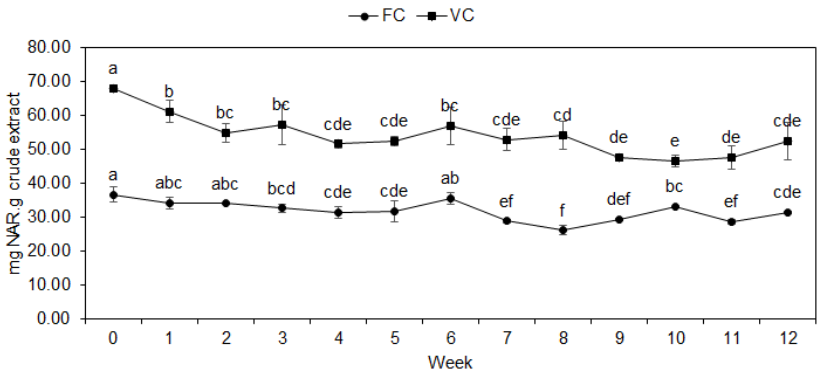

Figure 18 The effect of storage time on naringin of pomelo albedo's extract

A storage temperature at 8 °C was selected due to insignificant ($p > 0.05$) bioactive compound (naringin) observed at lower temperature (< 5°C) and frozen storage condition for storage of grapefruit juices (Igual et al., 2011). Naringin (NAR) is the most abundant flavonoids in grapefruit juice followed by narirutin (NAT), quercetin (QUER) and naringenin (NAG) (Igual et al., 2011). In the current study, pomelo albedo crude extract showed different values of naringin content after being dried by vacuum-dried (67.95 ~ 46.63 mg NAR/g crude extract) and freeze-drying (36.69 ~ 26.11 mg NAR/g crude extract) respectively. During initial week (Week 0), naringin was observed to be 67.95 mg NAR/g CE (28.52g NAR/kg dry based) (Table 4.12) after being treated with vacuum drying whereas 36.69 mg NAR/g CE (16.53g NAR/kg dry based) from freeze drying processing. These results are in line with the result reported by Pichaiyongvongdee and Haruenkit (2009) who reported 28.508 g naringin/kg dry weight in Kao Tanggkya pummelo varities from Thailand. Vacuum dried crude extract (VC) showed significant higher ($p < 0.05$) by 40% differences (Refer to Appendix D, Table D15) in the initial week in comparison with freeze dried crude extract (FC). This indicates that the naringin is sensitive to freeze drying. The results were consistent with previous study discovered freeze drying present lower value than fresh grapefruit during post-harvest (Vanamala et al., 2005).

Nevertheless, VC illustrated at 1st week showed significant reduction ($p < 0.05$) compared to FC which constantly did not show any significant difference ($p > 0.05$) till it reach week 7. After 1st week, the differences of VC showed significantly ($p < 0.05$) higher (~ 9.89%) in comparison with FC (~ 6.96%). It reflects the instability of VC and

causes liberation of naringin during storage period. During the first week until 3rd week, VC does not show any significant value/trend ($p > 0.05$), indicating the stability of naringin compound during storage, which is consistent with the presence of total phenolic content (Figure 18) value that is greater than in FC. This is due to the high thermal stability of naringin compound when treated with drying temperature lower than 100 °C. The findings from the research reported that naringin was sensitive to an exposure to visible light compared to oxygen and lower temperature (Ioannou et al., 2018). The experiment and storage of current study were conducted under non-visible light environment (lamp covered with infra-red plastic) and the samples were wrapped with covered container to avoid exposure to UV visible light. However, there was slight increase in value of VC during storage at week 3, nevertheless, insignificant value ($p > 0.05$) of NAR were observed. Thus, VC is considered still stable until week 9.

As for FC, there was significant increase ($p < 0.05$) in value of naringin content during week 6. Condensation of dried crude extract during storage indirectly increasing the value of bounded naringin. The temperature differences from the storage condition to room temperature could be the reason of condensation occurs when handling the samples. In addition, after 7th week of storage, the naringin compound of FC decreased significantly ($p < 0.05$) by 18.70%. The decreasing trend may due to the condensation of extracts on the surface of the container when exposed to atmospheric condition during handling of experiments.

4.5.4 Effect of storage time on antioxidant capacities of pomelo albedo's extract

The ability to scavenge free radical compound and ability to reduce ferric reducing of major compound across time was investigated during storage. Low degradation or high stability of the antioxidant capacities are preferred, and the current study reported on the comparison between control and treated samples during 12 weeks of storage. The rate of degradation was evaluated to determine the suitable order that suits the best trend of antioxidant capacities of pomelo albedo's extract during storage. Meanwhile, the current section focuses on the effects of storage time

on antioxidant capacities of pomelo albedo namely scavenging radical capacity; DPPH and ferric reducing ability power; FRAP.

4.5.4.1 Effects of storage time on DPPH of pomelo albedo's crude extract

Figure 19 displays scavenging radical ability (DPPH) by using expression of trolox equivalent of the stored crude extract affected by freeze dried and vacuum dried treatment.

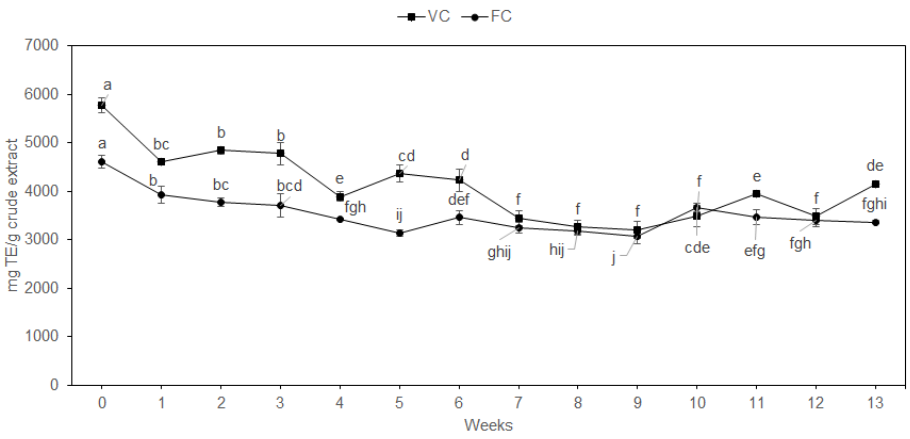

Figure 19 Degradation of scavenging radical activity **(**DPPH**)** of pomelo albedo's extract

In general, significant changes ($p < 0.05$) in the amount of DPPH scavenging ability were observed over time. The reason DPPH scavenging ability reduced across the time could be due to the presence of phenolic compounds. **Error! Reference source not found.** shows the results decreased significantly ($p < 0.05$) across time. After the 1st week of storage, the result of DPPH value reduces significantly ($p < 0.05$) in vacuum and freeze dried crude extract. This could be due to detrimental effect of heat on antioxidant compound present in VC compared to FC (Rafiq et al., 2019).

However, no significant changes ($p > 0.05$) were observed of scavenging radical ability (refer Appendix D, Table D17) during the first week till third week of storage time. In other words, the DPPH values for both dried treatments were practically constant till week 3 but reduced significantly ($p < 0.05$) over the range at week 4 by

18.6% and 7.9% differences for VC and FC respectively. It might be due to the limited presence of naringin compound (refer Figure 4.10, Week 4) in the product to scavenge the radical compound available. The trends of scavenging radical ability starts to tell apart between both treatments, VC or FC, whereby the DPPH values also decreased for week 5, while increasing value of DPPH was observed by VC. These indicates the liberation of complex polyphenols into low-molecular-weight antioxidant capacities containing compounds and formation of melanoidin like pigment during Maillard's reaction which are well known for their antioxidant activity (Rafiq et al., 2019).

4.5.4.2 Effects of storage time on FRAP of pomelo albedo's extract

Effects of storage period on pomelo albedo extract by freeze drying (FD) and vacuum oven drying (VD) are shown in Figure 20.

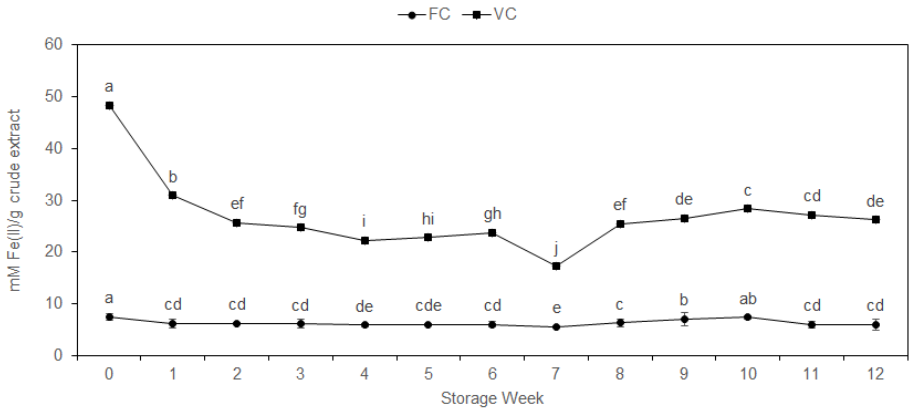

Figure 20 Degradation of inhibition of ferric ability (FRAP) of pomelo albedo's extract

FRAP indicates the reducing power ability of ferric which has been expressed by Ferrous (II) equivalent. In the albedo extract it was discovered that there is reduction of Ferric (III) to Ferrous (II). In general, results of VC showed they are within the range (48.30-17.24 Mm Fe (II)/g crude extract) and significantly higher ($p < 0.05$) concentration of FRAP value than FC (7.46 – 5.62 mM value) with the differences between VC and FC were approximately 84%. The results of FC showed from week 0 to week 1 was reduced with ~16% changes compared to the FRAP value of VC which has been reduced significantly ($p < 0.05$) by ~35%. Then, after 1st week, FC showed constant retention of antioxidant capacity till week 6. Meanwhile, VC continuously reduced after

1st week till week 7. However, during 8th week, the results of VC showed higher value of FRAP and becoming constant until week 12. This reflect the ferrous compound was retained longer in FC compared to VC compound. Furthermore, similar with VC, FC showed significantly increasing ($p < 0.05$) trend during 8th week and 9th week whereby these correlates well with the content of naringin FC during similar (refer Figure 4. 10) and reduced on 11th week. In this case, the increasing trend of FRAP value is due to the presence of phenolic indicating naringin from albedo extract that can reduce the ferric (III) compound.

The results obtained in this study showed that when the effect of drying process and storage is considered together, the application of vacuum drying process is attributed to a greater retention of all the analyzed phenolic, thereby representing a good alternative than freeze drying process (control).

4. 5. 5 Relationship between total phenolic content and naringin with antioxidant capacity during storage

Identification of relationship between naringin and total phenolic content with antioxidant activity of pomelo albedo were analyzed by Pearson's correlation (Table 38).

Table 38 Pearson's correlation between naringin with antioxidant activities (DPPH and FRAP) values.

Antioxidant content	**Antioxidant capacity**		
Analysis	**Total phenolic content (TPC)**	**DPPH free radical scavenging activity**	**Ferric Reducing Ability power (FRAP)**
Naringin (NAR)	r =0.946[a]	r = 0.622[a]	r = 0.910[a]
Total phenolic content (TPC)	-	r = 0.566[a]	r = 0.930[a]

r = pearson correlation

Positive correlation was observed between naringin compositions with DPPH free radical scavenging activity. It can be clearly seen that naringin is strongly correlated ($r = 0.622$) with DPPH value, which reflects the inhibition value of DPPH that might be indicated by the presence of naringin compound in pomelo albedo. It was statistically significant at 0.01 level. In addition, pomelo residues contain abundant phenolic compound an attribute and effect of DPPH free radical compound. Naringin also was found to be mainly responsible for the variation in the content of total phenolic content of pomelo albedo's extract.

The existence of other phenolic compounds such as hesperidin, neohesperidin, naringenin each of the compounds possesses antioxidant role. This is one of the reason that strong correlation ($r = 0.566$) effects of TPC content on inhibition of DPPH values were observed (Castro-Vazquez et al., 2016; Mat Zain et al., 2014). The findings were comparable with previous studies (refer section 0) which found positive correlation between TPC and DPPH value.

4.6 Total phenolic content, naringin, and antioxidant capacity of albedo's extract predicted by kinetic model- evaluation of reaction order and rate constant

4.6.1 Kinetic degradation of TPC content of pomelo albedo extracts during storage

For a proper comparison between two types of drying methods and their effects on selected antioxidant capacities, the results were adjusted to a zero order, first order and second order kinetics equation. The coefficients of determination (R^2) and the degradation constants (k) of each drying method on total phenolic content, naringin, DPPH and FRAP were evaluated. The order of reaction was estimated graphically by comparing the coefficients of determination (R^2) and standard error of estimate (SEE) obtained from plots of TPC content change as a function of storage time at similar condition storage temperature (Table 39).

Table 39 Comparison of reaction kinetic models and rate constants of TPC at different pre-treatment of drying condition

Kinetic model	Drying process	k (week^{-1})	R^2	SEE	$t_{1/2}$
$[TPC] = -kt + [TPC]_0$ n = 0	FC	-0.0241	0.4831	0.1180	20.7
	VC	-0.0255	0.8490	0.0484	19.6
$\ln[TPC] = -kt + ln[TPC]_0$ n = 1	FC	-0.0318	0.5150	0.1461	21.8
	VC	-0.0299	0.8654	0.0529	23.2
$\frac{1}{[TPC]} = kt + \frac{1}{[TPC]_0}$ n = 2	FC	0.0431	0.5517	0.1838	23.2
	VC	0.0352	0.8799	0.0584	28.4

k: rate constant of TPC degradation or TPC loss at 8 °C (week^{-1})

$t_{½}$: half-time

SEE: standard error estimate between experimental and predicted data

R^2: coefficient of determination

On the basis of the R^2 and SEE range values of the non-linear regression (of each types of pre-treated crude extract), the degradation of TPC from VC fits better with all models (zero, first and second) with R^2 values range of 0.8490 - 0.8799 and SEE range values of 0.0484 - 0.0584. Conversely, the R^2 value were much lower with FC extract (with R^2 range values of 0.4831 – 0.5517 and SEE range values 0.1180 – 0.1838), which confirmed that TPC degradation in different crude extract's dried methods could be varied and not fitted by overall kinetic reaction model (Table 4.13). The predictive values for TPC using zero order is y = 0.8037-0.0241x and y = 0.9703-0.0255x, while first order kinetic models of y = -0.2383-0.0318x and y = -0.0296 -0.0299x, and lastly second order present y = 1.2991 + 0.0431x and y = 1.0288 +0.0352x (where y is ln C (depends on order) and x is the storage time) from FC and VC, respectively. The related graph between experimental and predicted FC and VC based on order as in Figure 21.

On the contrary, mathematical models showed a poor fit for the curve particularly for freeze dried (FC) crude extract on TPC. Nevertheless, the first order model was used in most studies to describe the TPC degradation in extract of citrus during drying (Nesrine et al., 2015). Several studies have shown that citrus degradation

reactions follow the first- order model as degradation of anthocyanins, phenolic compounds, and antioxidant activity (Ghanem et al., 2018; Lago & Noreña, 2017; Remini et al., 2015).

The degradation rate constant (rate of TPC being degraded over time) is higher in VC compared to FC, and the difference is slightly significant ($p < 0.05$). The findings are presented in Table 39 and the table shows that the TPC degradation rate constant (k) reduced by 5.5% (zero order), increased by 6.0% (first order) and 18.33% (second order) between FC and VC respectively. This higher trend in degradation constant caused by vacuum drying reflects the preservation the heat composition during drying, resulting higher phenolic compounded in vacuum drying crude during storage.

As shown in Figure 21, the change of TPC content decreased according to the time for both crude extracted pre-treated by freeze drying and vacuum drying. The longer the storage time, the greater the degradation of TPC. Approximately mean average of 41.78% and 24.74% of initial TPC content remained after 12 weeks of storage at 8°C (refrigerated conditions) in FC and VC, respectively. In brief, drying process may result in high or low levels of TPC depending on the type of phenol compounds present in the plant material (Ghanem et al., 2018). In the current findings, drying by vacuum oven (VC) on TPC fitted well with first order kinetic model. Consistent with previous study reported by Ghanem et al. (2018) that found the lemon by-product fitted well by a first-order kinetic model to applied microwave drying methods on total flavonoid content.

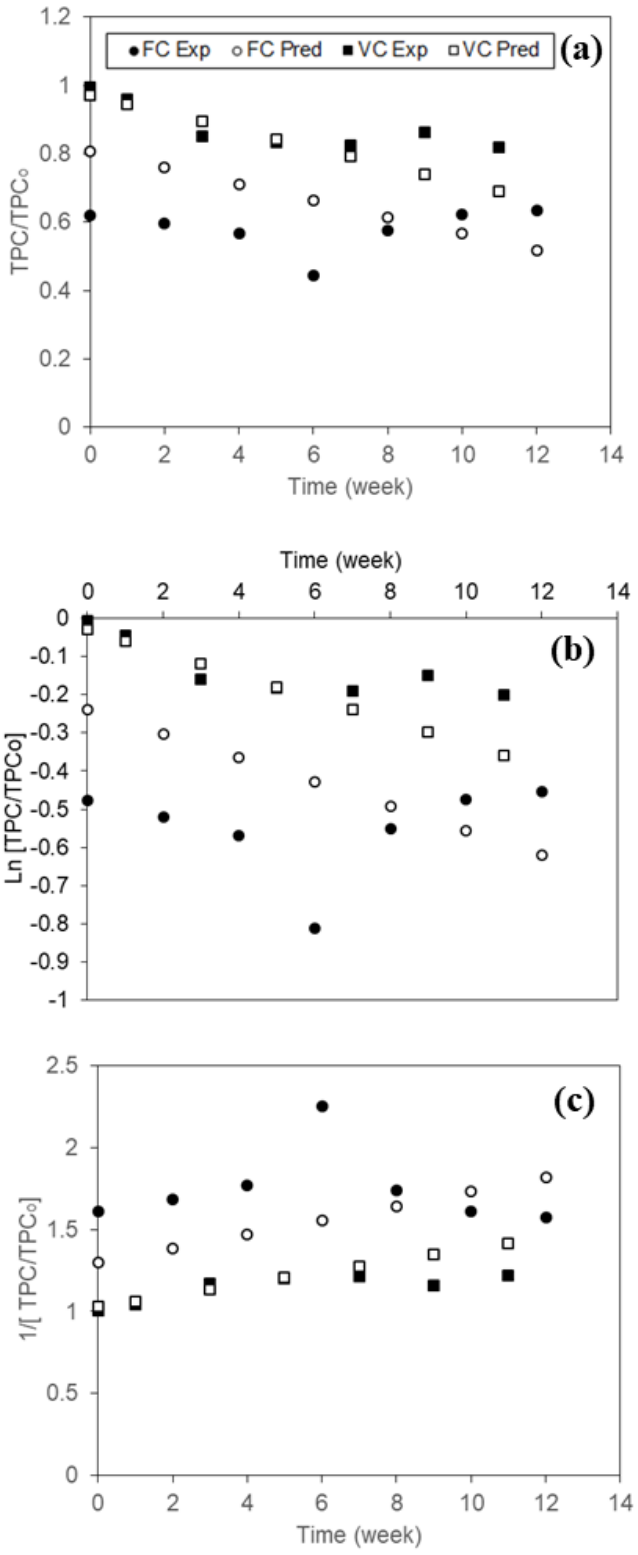

Figure 21 Degradation kinetics of TPC (a) Zero order (b) First order (c) Second order**:** freeze**-**dried crude (FC) extract and Vacuum dried crude (VC) extract

Half time through TPC degradation from the initial concentration were compared between kinetic orders and variation of the $t_{1/2}$ value was observed between FC and VC. The value of $t_{1/2}$ were varied from 21 to 23 weeks for FC whereas 20 to 28 weeks discovered between the kinetic orders (zero, first and second order). Higher $t_{1/2}$ were discovered in second order reaction for FC (23 weeks) and VC (28 weeks) respectively. This result shows longer period of VC to degrade to half of the initial concentration compared to FC.

In addition, the predicted VC extract was highly comparable with the experimental ones which conforms to the results of higher R^2 than FC extract in overall order.

4.6.2 Kinetic degradation of naringin of pomelo albedo extracts during storage

Table 40 depicts the kinetic parameters k, half time ($h_{½}$) as well as the R^2, R^2 adj , standard error estimate (SEE) of zero, first and second order model through a least square fitting procedure of the naringin degradation on FC and VC extracts. As can be seen, a good fit was obtained by plotting the graph using Arrhenius equation ($R^2 > 0.89$) and (SEE < 0.05) for VC similar to FC which displays R^2 more than 0.89 and SEE value was less than 0.04.

Table 40 Comparison of reaction kinetic models and rate constants at different pre-treatment of drying condition on naringin compound

Kinetic model	Drying process	Kinetic parameter			
		k (week^{-1})	R^2	SEE	$t_{1/2}$
$[NAR] = -kt + [NAR]_0$ n = 0	FC	-0.0191	0.8916	0.0298	26.2
	VC	-0.0252	0.8939	0.0390	19.8
$\ln[NAR] = -kt + ln[NAR]_0$ n = 1	FC	-0.0219	0.9024	0.0323	31.6
	VC	-0.0306	0.9162	0.0415	22.6
$\frac{1}{[NAR]} = kt + \frac{1}{[NAR]_0}$ n = 2	FC	0.0252	0.9106	0.0354	39.7
	VC	0.0375	0.9326	0.0452	26.7

k: rate constant of NAR degradation or NAR loss at 8 °C (week^{-1})

$t_{½}$: half-time

SEE: standard error estimate between experimental and predicted data

R^2: coefficient of determination

n indicates the order of reaction. Equation 1, 2 and 3 represent zero order, first order, and second order model respectively.

The degradation rate constant (k) was lower in VC (-0.0252) compared to FC (-0.0191). Under vacuum drying methods condition, the k constants in VC extracts were shown -2.52 x 10^{-2} week^{-1}, -3.06 x 10^{-2} week^{-1} and 3.75 x 10^{-2} week^{-1} for zero, first and second order respectively. Meanwhile, the value of k constant was observed from zero order (-1.91 x 10^{-2} week^{-1}), first order (-2.19 x 10^{-2} week^{-1}) and second order (2.52 x 10^{-2} week^{-1}) which reflected the higher stability of naringin compound in freeze dried crude extract against vacuum dried crude extract. Regardless of the lower concentration of naringin compound in the freeze-dried crude extracts (Table 40), the degradation of naringin attained a slower rate degradation compared to vacuum drying modes. Freeze drying is an effective drying method to retain naringin compound in pomelo albedo.

Even though, there was not much difference between both drying methods, the half-life time were compared directly and showed significant difference ($p < 0.05$) between kinetic models. In terms of zero order, the results clearly show that naringin

from FC take longer time (40 days) to be degraded in comparison to VC (27 days). The results demonstrated that freeze drying process resulted in higher stability dried extracts in comparison to vacuum drying process with a difference in the $t_{1/2}$ value up to 11 days during storage. Freeze drying retain the dry extract (naringin) in frozen state prior to drying promote that limited the exposure of oxygen which could promote or degrade naringin compared to VD.

Nevertheless, Ioannou et al. (2018) found that kinetic of naringin degradation was modelled following a first-order reaction during heat treatment (100,110, 120 and 130 ℃). It can be explained by the presence of glycosyl compound in naringin structures. Estimated value of energy activation (100.6 kJ/ mol) were observed by Ioannou et al. (2018) to degrade the naringin structure, however, with an additional presence of the glycosyl groups like rutins, higher requirement of activation energy (107.3 kJ/mol) were needed. Thus, naringin could not simply degrad during storage particularly in dried extract form. Thus, according to Figure 22, most of experimental data fit into the predicted data by zero, first and second order respectively.

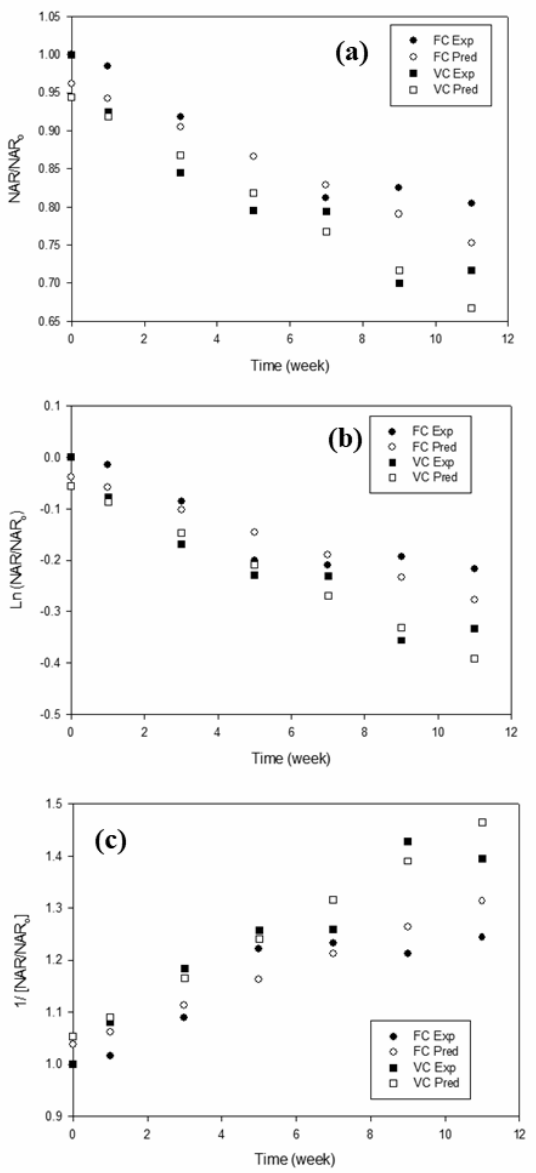

Figure 22 Degradation kinetics of naringin based on (a) Zero order (b) First order (c) Second order**:** freeze-dried crude (FC) and Vacuum dried crude (VC) extract

4.6.3 Kinetic degradation of scavenging radical DPPH of pomelo albedo extract during storage

The degradation rate of DPPH radical scavengers of the VC and FC were calculated using equation 3.26 - 3.27 and the results are summarized in Table 41. Thus, comparison of kinetic models and rate constant for different kinetic order were also was evaluated in similar table.

Table 41 Comparison of reaction kinetic models and rate constants at different pre-treatment of drying condition on DPPH scavenging radical

Kinetic model	Drying process	Kinetic parameter			
		k (week^{-1})	R^2	SEE	$t_{1/2}$
$[DPPH] = -kt + [DPPH]_0$ n = 0	FC	-0.0219	0.5779	0.0839	22.83
	VC	-0.0317	0.7668	0.0827	15.77
$\ln[DPPH] = -kt + ln[DPPH]_0$ n = 1	FC	-0.0267	0.5756	0.103	25.96
	VC	-0.0421	0.7796	0.106	16.46
$\frac{1}{[DPPH]} = kt + \frac{1}{[DPPH]_0}$ n = 2	FC	0.033	0.5671	0.1293	30.30
	VC	0.0572	0.7803	0.1435	17.48

k : rate constant of DPPH degradation or TPC loss at 8 °C (week^{-1})

$t_{½}$: half-time

SEE : standard error estimate between experimental and predicted data

R^2 **:** coefficient of determination

n indicates the order of reaction. Equation 1, 2 and 3 represent zero order, first order, and second order model respectively.

Table 41 summarizes the kinetic models and rate constant on DPPH for dried crude extract of albedo. The results presented indicates degradation of DPPH for VC which were slightly higher by 30.9%, 36.6% and 42.3% for zero, first and second order than FC respectively. Based on vacuum drying condition, the rate constant in VC extracts presents -3.17 x 10^{-2} week^{-1}(zero order), -4.21 x 10^{-2} week^{-1} (first order) and 5.72 x 10^{-2} week^{-1} (second order) respectively compared to in zero order (-2.19 x 10^{-2} week^{-1}), first order (-2.67 x 10^{-2} week^{-1}) and second order (3.30 x 10^{-2} week^{-1}) of FC. The rate constant of VC were higher than FC in overall order signifies the rate of degradation on DPPH were faster in VC compared to FC. Consistent with TPC findings in Table 4.13, the value of R^2 of VC which corresponds to DPPH degradation were between the ranges (0.77 - 0.78) while SEE values were between the range of 0.09 to 0.14.

In contrast with FC, the R^2 values were in the range of 0.57 - 0.58 while SEE values were between of 0.08 to 0.13. In brief, the trend of DPPH degradation of VC fits

better with zero order kinetic reaction with R^2 value (0.77) and lower SEE value (0.083). In addition, predicted at half time for the ability of scavenging, DPPH radical were reduced between FC and VC at different kinetic models reaction. Similar with previous findings on TPC, where the higher value of t ½ was shown in FC compared to VC. Nevertheless, the half time in DPPH (23 weeks) results seems higher with t ½ value for TPC (21 weeks) at zero order reaction, and retained longer for the first order (22 weeks) and second order (23 weeks) model for DPPH value in comparison to TPC (25 and 30 weeks for first and second order respectively) for FC. Lower value was shown in VC whereby the half-life of the DPPH was within the range of 16-17 weeks only.

In addition, Figure 23 reflects the predicted and experimental of DPPH scavenging radical of FC and VC for zero, first and second order reaction. The trend of both pre-treated were differed with predicted ones regardless of reaction kinetic models (zero, first and second order models). Zero order were found to be parallel with predicted ones compared to other order, indicating zero order can be used to predict the behaviour of FC and VC on DPPH scavenging.

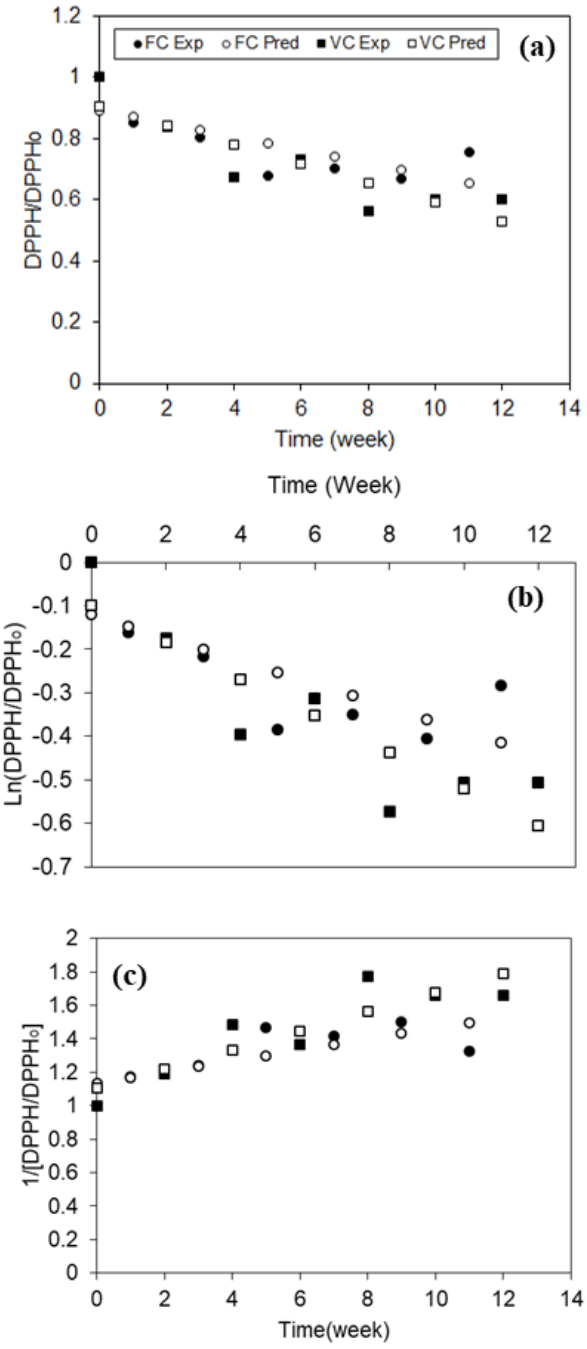

Figure 23 Degradation kinetics of experimental and predicted of freeze-dried crude extract (FC) and Vacuum dried crude extract (VC) on DPPH scavenging radical based on (a) Zero order (b) First order (c) Second order

4.6.4 Kinetic of FRAP reduction of pomelo albedo extract during storage

Different kinetic models (zero order, first order, and second order) for each pre-treated drying method (freeze-drying and vacuum drying) were tabulated in Table 42.

Table 42 Comparison of reaction kinetic models and rate constants at different pre-treatment of drying condition on FRAP ability

Kinetic model	Drying process	Kinetic parameter			
		k (week^{-1})	R^2	SEE	$t_{1/2}$
$[FRAP] = -kt + [FRAP]_0$ n = 0	FC	-0.0154	0.6842	0.0471	32.47
	VC	-0.0257	0.3465	0.1588	19.46
$\ln[FRAP] = -kt + ln[FRAP]_0$ n = 1	FC	-0.0178	0.7001	0.0522	38.93
	VC	-0.0351	0.3363	0.2212	19.74
$\frac{1}{[FRAP]} = kt + \frac{1}{[FRAP]_0}$ n = 2	FC	0.0205	0.7121	0.0586	48.78
	VC	0.0492	0.3154	0.3255	20.33

k: rate constant of FRAP degradation at 8 °C (week^{-1})

$t_{½}$: half-time

SEE: standard error estimate between experimental and predicted data

R^2: coefficient of determination

n indicates the order of reaction. Equation 1, 2 and 3 represent zero order, first order, and second order model respectively.

The constant rate of FRAP degradation value of FC (-1.54 x 10^{-2} week^{-1} to 2.05 x 10^{-2} week^{-1}) and VC (-2.57 x 10^{-2} week^{-1} to 4.92 x 10^{-2} week^{-1}) in overall kinetic order which are lower than in DPPH (Table 4.15). Nevertheless, the value of R^2 is lower than 0.75 and within the range for FC (0.68 -0.71) and VC (0.32 – 0.35) whereas the SEE is higher in VC (0.16 - 0.32) and FC (0.05 - 0.06). Thus, it can be concluded that reduction value of FRAP relatively fit into the selected kinetic models particularly for FC involved in the present study as lower R^2 value and high value of error estimate were observed.

In addition, Figure 24 shows comparison of experimental and predicted of FRAP value at different kinetic models order. However, the order of kinetic models fit better

in FC compared to VC for degradation of FRAP during storage. This is consistent with the lower value R^2 obtained by VC in Table 4.16 and higher SEE value. Nevertheless, VC extract might fit to other kinetic models such as Weibull model, Eyring model which has been discovered for the ascorbic acid degradation during storage (Remini et al., 2015).

In general, TPC assay can be viewed as a functional indicator of nutritional and quality deterioration of pomelo albedo extract after processing and subsequent storage, as this method had the similar trend (first order) which correlated well with specific compound namely naringin and antioxidant capacities (DPPH and FRAP). Different drying methods (FC and VC) do affect the degradation rate of TPC and naringin during storage and it corresponded well with antioxidant capacities. By observing the higher value coefficient of determination (R^2) and lower value of SEE between different reaction kinetic models and different parameters of quality assessment, significant variation ($p < 0.05$) were observed between FC and VC. The results in the present study illustrated that proper selection of kinetic model is critical to understand the antioxidant compound and capacities during degradation process. The degradation of TPC can be described in overall order (zero, first and second order) particularly in VC, while the highest R^2 were found in second order reaction. This is consistent with half-life reaction of VC which displayed the highest value of R^2.

Meanwhile, naringin degradation ($R^2 > 0.8$ and SEE >0.05) during both FC and VC were followed with zero, first and second order kinetic reaction. Naringin were found as the major compound observed in pomelo albedo extracts regardless of pre-treated by different drying methods. This compound was degraded during storage with lower rate of constant (less than 0.1) which correspond well with antioxidant capacities such as radical scavenging of DPPH. DPPH scavenger of VC extract followed zero order kinetic reaction with the half time of DPPH degraded approximately around 16 weeks. Meanwhile, the experimental analysis for FRAP (FC) reducing were found to give higher R^2 value (minimum value of 0.68) than VC (minimum value 0.32) within zero, first and second order models. Generally, the results of the kinetic parameters showed that TPC, naringin, DPPH and FRAP varied during pre-treated processing.

The findings may be of great help in predicting and understanding the changes of phenolic compounds (particularly naringin) during storage as well as antioxidant capacities of pomelo albedo extracts.

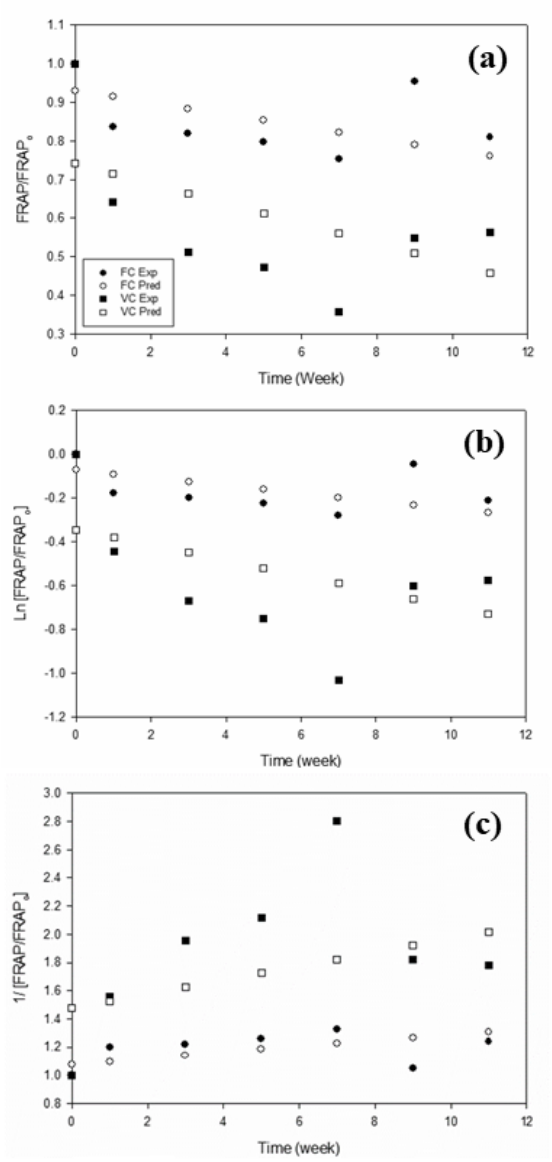

Figure 24 Degradation kinetics of FRAP ability based on (A) Zero order (B) First order (C) Second order**:** freeze**-**dried crude (FC) and Vacuum dried crude (VC); during storage at 8 ℃

CHAPTER 5
CONCLUSION AND FUTURE RECOMMENDATIONS

5.1 Conclusion

The selection of the pomelo residue to be further analyzed is based on the amount of total phenolic content. Higher phenolic compound has been used as a core parameter to differentiate the potential source of phenolic compound because phenolic compounds contain secondary metabolite structure which can scavenge the free radical and exhibiting variety of roles with therapeutic function to human health. Albedo was found to be containing the highest total phenolic content (TPC) followed by flavedo, lamella and pulp waste. Subsequently, the pomelo residue (albedo) chosen was analyzed by undergoing the selection of ideal drying condition between CD and VD. The target responses were based on; lowest MC, high TPC and high DPPH using factorial design from response surface methodology (RSM) for the selected pomelo residues. Thus, the selection of pomelo residues in the current study known as albedo, the optimum drying condition of pomelo albedo to retain antioxidant activity using vacuum drying at 90 ℃ with index ($D \geq 0.8$).

Next, identification of the best drying models which best describe the mechanism during drying process was based on higher R^2 and lowest standard error estimate (SEE) for different drying methods at overall temperature (50 – 90 ℃). During drying, the moisture removal undergoing diffusion approach is described as effective moisture diffusivity (D_{eff}). Energy activation (E_a) was compared at different drying temperature between CD and VD.

The second objective of the study focused on the identification of potential models which can be described during respective drying process. For conventional drying, diffusion approach models described the best in terms of mechanism drying model with high R^2 (>0.99) value and low value of SEE (< 0.24) and RMSE (< 0.021) were recorded. The range of D_{eff} was from 5.00×10^{-7} to 6.98×10^{-7} m^2/s for CD. The VD methods were best described using Two-term exponential model with high value

of R^2 (>0.99) and with low value of SEE (0.0117 - 0.0361) and RMSE (0.076 - 0.0295). The values of D_{eff} within the range of 9.86×10^{-7} to 1.30×10^{-6} m^2/s were observed. Each model containing several parameters can affect the expected results. The current study reported on correlation of specific parameter involved within the respective models.

Finally, the storage of crude extract at 8 ℃ for 12 weeks were analyzed and modeled to identify the changes of the TPC, phenolic compound and antioxidant properties. Naringin were found to exhibit higher stability in pomelo albedo extracts even though they had been pre-treated by different drying methods. Lower rate constant (less than 0.1) degradation of naringin was observed during storage which corresponds well with antioxidant capacities such as radical scavenging of DPPH and FRAP. DPPH scavenger of VC extract followed zero order kinetic reaction with the half time of DPPH degraded approximately around 16 weeks. Nevertheless, FC extract were found to be following zero, first and second order with higher value R^2 (0.68) better than VC (R^2 ~ 0.32). In brief, the findings of the kinetic parameters showed that TPC, naringin, DPPH and FRAP were varied during pre-treated processing. The data obtained from the current studies could be a great help in predicting the storage period for variation changes of phenolic (naringin) and its relationships with pomelo albedo extract. In conclusion, post-harvest agriculture, nutraceutical or food industrial sectors may find this study beneficial as a general guide for kinetics modeling and the effects of drying methods on characterization (physicochemical, nutritional composition, antioxidant capacities) of pomelo residues.

5.2 Future Recommendations

As the future recommendation, in terms of the product, different parts of pomelo residues could be a potential source for animal-feeds or nutraceutical supplements due to higher nutritional composition. In addition, characterization of combination of pomelo wastes can also be further explored which might contribute to higher nutritional composition. Different solvent extraction to obtain the highest

yields of bioactive compound related in different parts could also be further analysed. In addition, pre-treatment such as dipping sample into sodium chloride solution has been shown by previous studies it can sustain better quality based on osmotic dehydration process.

The effects of hybrid drying process on naringin include different drying methods (microwave freeze drying; vacuum infrared drying) for the pomelo residue could be studied. In addition, the rate of changes in quality (moisture ratio, color changes, total phenolic content, DPPH) during drying process, could also be further investigated in order to determine the rate of reaction. This phenomenon is significant whereby it can be used to predict the variations of quality observed during processing.

In terms of storage, the shelf life of a product value depends on storage life. The higher the stability of the product, the longer the bioactive compound can still exhibit positive antioxidant capacities. During storage, temperature, oxygen, and lightness might be the major factors that can affect the quality of end product. Different conditions of storage in different temperature and the effects of storage temperature on the quality of the final product can be investigated in the future.

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APPENDICES

A. Schematic diagram of freeze drying, conventional oven drying and vacuum oven drying methods

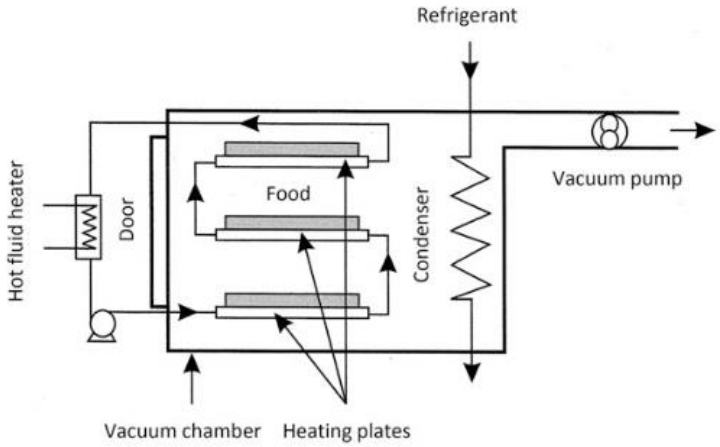

Figure A1: Schematic of freeze dryer.

Source: Reis (2014)

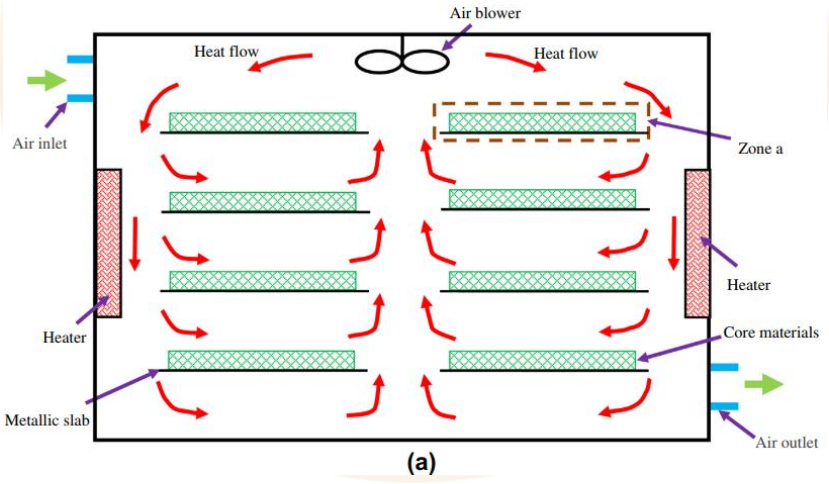

Figure A2: Schematic drawing of conventional hot air oven drying.

Source: Li et al. (2013)

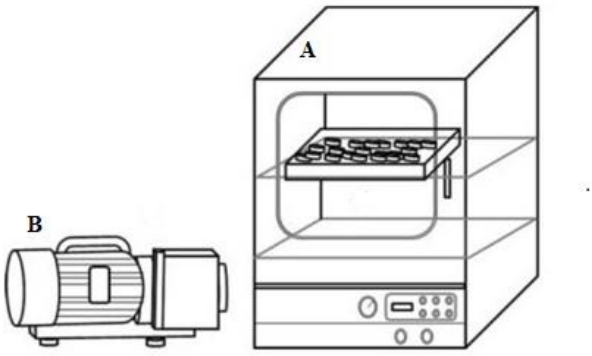

Figure A3. Schematic diagram of vacuum drying used in the study. A. Vacuum oven consisting of a temperature control unit, a vacuum gauge, a heating chamber, B. Vacuum pump.

[Source: Lee and Zuo (2013)]

B. Color (L*, a*, b*) of fresh pomelo residues

Table B1: Effects of different drying treatment and temperature on color of fresh pomelo residues

Treatment	L*	a*	b*
Flavedo	61.83 ± 1.58	-7.87 ± 0.22	36.98 ± 1.32
Albedo	76.41 ± 0.33	5.79 ± 0.33	21.09 ± 0.34
Lamella	67.58 ± 0.07	8.81 ± 0.16	24.66 ± 0.49
Pulp waste	53.01 ± 2.23	3.7 ± 0.67	24.87 0.79

C. Summary of analysis methods in table form

Table C1: Summary of the analysis methods involve samples related

Section	Analysis involved	Sample form	Responses
Stage 1	Physicochemical, nutritional composition, and antioxidant analysis of pomelo residues	Dried samples(flavedo, albedo, lamella, pulp waste)	Moisture content, selected nutritional composition (ash, protein and fat content), antioxidant properties (TPC, DPPH, FRAP)
	Selection of ideal drying condition using factorial design, desirability index	Dried samples-albedo	High TPC, Low MC High DPPH
Stage 2	-Modeling of drying kinetics, effective diffusivities(D_{eff}) and energy activation (E_a)	Fresh sample-Albedo	High coefficient determination, (R^2), low standard estimate error (SEE), Root mean square error (RMSE)
Stage 3	-Phenolic identification and storage analysis based on TPC, naringin and antioxidant capacity -Kinetic modeling using zero, first and second order	Crude extract of albedo	High coefficient determination, (R^2), low standard estimate error (SEE)

D. Results of statistical analysis

Table D1 Duncan table of composition per pomelo fruits.

Composition_per_fruit

	Sample	N	Subset for alpha = 0.05		
			1	2	3
Duncan[a]	Lamella	5	8.0540		
	Flavedo	5	8.2200		
	Pulp waste	5		16.3920	
	Albedo	5		20.2040	
	Pulp	5			62.5980
	Sig.		.937	.079	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 5.000.

Table D1.1 Duncan table of compositions per total residues.

Composition_per_total_residues

	Sample	N	Subset for alpha = 0.05		
			1	2	3
Duncan[a]	Lamella	5	15.3920		
	Flavedo	5	15.5620		
	Pulp waste	5		31.2800	
	Albedo	5			37.7640
	Sig.		.947	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 5.000.

Table D2 Duncan table for MC affected by different drying methods on Flavedo.

Moisture Content

	Sample	N	Subset for alpha = 0.05							
			1	2	3	4	5	6	7	8
Duncan[a]	Flavedo CD80	3	4.0467							
	Flavedo CD90	3		4.8467						
	Flavedo CD60	3		5.1467	5.1467					
	Flavedo VD90	3			5.7129					
	Flavedo VD80	3				6.5060				
	Flavedo CD70	3				6.8067				
	Flavedo VD70	3					8.6638			
	Flavedo FD	3					9.3300			
	Flavedo CD50	3						10.1833		
	Flavedo VD60	3						10.8026		
	Flavedo VD50	3							16.6033	
	Fresh Flavedo	3								80.5933
	Sig.		1.000	.406	.124	.405	.073	.094	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 3.000.

Table D3 Duncan table for MC affected by different drying methods on albedo

Moisture content

	Sample	N	Subset for alpha = 0.05							
			1	2	3	4	5	6	7	8
Duncan[a]	Albedo CD70	3	3.3267							
	Albedo CD80	3	3.7167							
	Albedo CD90	3		4.6433						
	Albedo CD60	3			5.8800					
	Albedo CD50	3			6.5333					
	Albedo VD80	3				7.5831				
	Albedo VD90	3					8.5654			
	Albedo FD	3					8.6300			
	Albedo VD70	3					8.9479	8.9479		
	Albedo VD60	3						9.7162		
	Albedo VD50	3							20.2567	
	Fresh Albedo	3								77.6900
	Sig.		.338	1.000	.114	1.000	.375	.066	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 3.000.

Table D4 Duncan table for color (L) affected by different drying methods on flavedo.

L

	Colour Parameter	N	Subset for alpha = 0.05							
			1	2	3	4	5	6	7	8
Duncan[a]	Flavedo VD60	3	37.7033							
	Flavedo VD90	3	38.4367	38.4367						
	Flavedo VD50	3	38.7033	38.7033						
	Flavedo VD80	3		39.7367						
	Flavedo VD70	3			41.7367					
	Flavedo CD90	3				43.7000				
	Flavedo CD60	3					54.3333			
	Flavedo CD70	3						55.7667		
	Flavedo CD80	3							57.7333	
	Flavedo CD50	3							58.9333	58.9333
	Flavedo FD	3								59.6333
	Sig.		.144	.061	1.000	1.000	1.000	1.000	.068	.275

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 3.000.

Table D5 Duncan table for color (b) affected by different drying methods on flavedo.

b

	ColourParameter	N	Subset for alpha = 0.05				
			1	2	3	4	5
Duncan[a]	Flavedo VD50	3	27.0000				
	Flavedo VD60	3	27.2667				
	Flavedo VD70	3		29.4333			
	Flavedo VD80	3		29.5000			
	Flavedo VD90	3		29.5000			
	Flavedo CD90	3			34.5000		
	Flavedo CD60	3			34.6333		
	Flavedo FD	3				36.8667	
	Flavedo CD50	3				37.0333	
	Flavedo CD70	3					38.5333
	Flavedo CD80	3					39.3333
	Sig.		.507	.875	.739	.678	.056

Means for groups in homogeneous subsets are displayed.
a. Uses Harmonic Mean Sample Size = 3.000.

Table D6 Duncan table for ash content affected by different drying methods on flavedo.

AshContent

	Sample	N	Subset for alpha = 0.05				
			1	2	3	4	5
Duncan[a,b]	Flavedo VD50	2	5.8764				
	Flavedo FD	3	5.9200	5.9200			
	Flavedo VD90	2	6.2940	6.2940	6.2940		
	Flavedo VD80	2		6.4282	6.4282		
	Flavedo VD70	2		6.4402	6.4402		
	Flavedo CD50	2		6.4500	6.4500	6.4500	
	Flavedo CD90	2			6.5000	6.5000	
	Flavedo VD60	2			6.5238	6.5238	
	Flavedo CD60	2			6.7500	6.7500	6.7500
	Flavedo CD80	2				7.0000	7.0000
	Flavedo CD70	2					7.1500
	Sig.		.110	.058	.102	.050	.125

Table D7: Duncan table for ash content affected by different drying methods on albedo

Ashcontent

	Sample	N	Subset for alpha = 0.05			
			1	2	3	4
Duncan[a,b]	Albedo FD	3	2.6367			
	Albedo VD60	2	3.1500	3.1500		
	Albedo VD50	2	3.2650	3.2650		
	Albedo VD90	2	3.2950	3.2950		
	Albedo VD70	2	3.3050	3.3050		
	Albedo CD70	2	3.3500	3.3500		
	Albedo VD80	2	3.4550	3.4550		
	Albedo CD60	2		3.6000	3.6000	
	Albedo CD90	2		3.9000	3.9000	3.9000
	Albedo CD80	2			4.4000	4.4000
	Albedo CD50	2				4.6000
	Sig.		.063	.086	.054	.086

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.063.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Table D8 Duncan table for ash content affected by different drying methods on lamella

Ash Content

	Sample	N	Subset for alpha = 0.05			
			1	2	3	4
Duncan[a,b]	Lamella FD	3	2.2867			
	Lamella VD(50)	2	2.5650	2.5650		
	Lamella CD(90)	2	2.6500	2.6500	2.6500	
	Lamella VD(70)	2	2.7700	2.7700	2.7700	
	Lamella VD(90)	2		2.9020	2.9020	
	Lamella VD(60)	2		2.9400	2.9400	
	Lamella CD(60)	2		3.0500	3.0500	3.0500
	Lamella CD(70)	2		3.0500	3.0500	3.0500
	Lamella VD(80)	2		3.1250	3.1250	3.1250
	Lamella CD(80)	2			3.2500	3.2500
	Lamella CD(50)	2				3.6500
	Sig.		.105	.075	.059	.054

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.063.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Table D9 Duncan table for ash content affected by different drying methods on lamella

Ashcontent

	Sample	N	Subset for alpha = 0.05				
			1	2	3	4	5
Duncan[a,b]	Pulp waste VD60	2	3.1400				
	Pulp waste FD	3	3.2333	3.2333			
	Pulp waste VD50	2	3.3850	3.3850	3.3850		
	Pulp waste VD80	2	3.5000	3.5000	3.5000		
	Pulp waste VD70	2	3.5250	3.5250	3.5250		
	Pulp waste VD90	2	3.5350	3.5350	3.5350		
	Pulp waste OD80	2		3.9500	3.9500		
	Pulp waste OD90	2			4.0500		
	Pulp waste OD70	2				5.0000	
	Pulp waste OD60	2				5.0500	
	Pulp waste OD50	2					10.6500
	Sig.		.295	.071	.091	.881	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.063.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Table D10 Duncan table for protein content affected by FD on pomelo residues

Protein_content

	Sample	N	Subset for alpha = 0.05			
			1	2	3	4
Duncan[a]	Albedo FD	2	3.4908			
	Lamella FD	2		4.8773		
	Flavedo FD	2			7.6302	
	Pulp Waste FD	2				8.1769
	Sig.		1.000	1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.000.

Table D11 Total phenolic content affected by freeze drying, conventional drying and vaccum drying methods of different parts of pomelo residues (flavedo, albedo, lamella, pulp waste) at different drying temperature (50-90°C).

Treatment	mg GAE/100g DW			
Sample	Flavedo	Albedo	Lamella	Pulp waste
FD	1045.14 ± 7.88e	1267.87 ± 16.54f	942.49 ± 4.31g	554.65 ± 8.84e
CD50	1225.69 ± 2.05d	1330.33 ± 12.51f	986.46 ± 19.17g	961.92 ± 15.37b
CD60	1167.34 ± 18.85de	1336.77 ± 16.51f	997.61 ± 8.73g	892.56 ± 3.08c
CD70	2015.30 ± 125.55b	2007.46 ± 41.38d	1867.90 ± 48.69b	926.49 ± 4.85bc
CD80	1955.95 ± 7.86b	2370.10 ± 23.89c	1961.78 ± 34.52a	770.37 ± 56.62d
CD90	3016.27 ± 178.40a	2925.63 ± 27.46a	1860.43 ± 58.58b	1387.42 ± 72.42a
VD50	1281.82 ± 11.87d	1790.94 ± 42.84e	1354.58 ± 31.18e	529.48 ± 20.60e
VD60	1559.08 ± 87.75c	2030.48 ± 65.30d	1467.38 ± 39.08d	569.66 ± 29.08e
VD70	1609.71 ± 36.23c	2099.18 ± 83.43d	1571.29 ± 20.59c	254.20 ± 6.87h
VD80	1610.71 ± 39.50c	2608.42 ± 67.15b	1616.77 ± 41.81c	352.83 ± 37.05g
VD90	1948.26 ± 94.99b	2516.51 ± 41.70b	1917.11 ± 41.33ab	507.78 ± 48.40e

The results signifies the mean value ± standard deviation with different letters in the **same column** indicate that the values are significantly different ($p < 0.05$) between different drying treatment; FD: Freeze drying; CD: Conventional drying; VD: Vacuum drying

Table D12 Effects of drying methods on DPPH scavenging activity of pomelo residues at different drying temperature.

Sample	Flavedo	Albedo	Lamella	Pulp waste
FD	86.21 ± 0.53b	93.03 ± 0.49a	92.32 ± 0.36a	90.47 ± 0.84a
CD50	87.92 ± 0.27a	92.25 ± 0.84a	92.01 ± 0.44a	80.28 ± 2.31c
CD60	85.03 ± 0.38bc	89.26 ± 0.27b	90.43 ± 0.62b	85.14 ± 0.40b
CD70	82.36 ± 0.34d	69.24 ± 2.17g	80.43 ± 0.71d	81.42 ± 0.18c
CD80	74.96 ± 0.29f	78.71 ± 0.11e	79.47 ± 0.11de	81.09 ± 0.22c
CD90	47.00 ± 1.49g	48.01 ± 2.97h	67.91 ± 0.44h	67.79 ± 0.10e
VD50	80.56 ± 0.44e	79.75 ± 0.97e	67.33 ± 0.67h	66.46 ± 1.24e
VD60	79.75 ± 0.98e	82.70 ± 0.32d	72.57 ± 1.79g	71.86 ± 1.77d
VD70	84.73 ± 0.09c	74.61 ± 0.57f	78.15 ± 0.66ef	80.95 ± 0.81c
VD80	84.86 ± 0.05c	80.49 ± 0.74e	81.89 ± 0.27c	81.30 ± 0.22c
VD 90	82.29 ± 0.11d	85.46 ± 0.30c	77.33 ± 0.86f	81.48 ± 0.51c

Different letters in the **same column** indicate that the values are significantly different ($p < 0.05$)

Table D13 Effects of drying methods on FRAP of pomelo residues at different drying temperature

Treatment	mM Fe (II)/g DW			
Sample	Flavedo	Albedo	Lamella	Pulp waste
FD	4.00 ± 0.06c	2.56 ± 0.10f	1.99 ± 0.03h	3.07 ± 0.10d
OV50	2.61 ± 0.04fg	4.57 ± 0.19c	1.89 ± 0.05h	2.84 ± 0.05de
OV60	3.69 ± 0.01cd	3.86 ± 0.08d	2.63 ± 0.05g	5.22 ± 0.09b
OV70	3.07 ± 0.05ef	2.05 ± 0.09g	2.69 ± 0.11fg	2.90 ± 0.05de
OV80	5.23 ± 0.11b	4.93 ± 0.09b	4.10 ± 0.08b	3.11 ± 0.13d
OV90	13.96 ± 0.94a	11.33 ± 0.27a	5.78 ± 0.16a	18.81 ± 0.58a
VD50	2.40 ± 0.08g	1.78 ± 0.02h	1.72 ± 0.02i	1.31 ± 0.03g
VD60	3.40 ± 0.31de	4.82 ± 0 17b	1.91 ± 0.04h	1.74 ± 0.06f
VD70	3.35 ± 0.07de	1.98 ± 0.03gh	2.97 ± 0.11e	2.68 ± 0.20e
VD80	4.00 ± 0.18c	2.38 ± 0.08f	2.78 ± 0.04f	2.88 ± 0.03de
VD90	4.87 ± 0.22b	3.47 ± 0.11e	3.73 ± 0.07c	4.03 ± 0.19c

The results signifies the mean value ± standard deviation with different letters in the **same column** indicate that the values are significantly different ($p < 0.05$) between different drying treatment; FD: Freeze drying; CD: Conventional drying; VD: Vacuum drying

Table D14 Duncan table for protein content affected by FD on pomelo residues

DPPH

	Sample	N	Subset for alpha = 0.05				
			1	2	3	4	5
Duncan[a]	VD50 Pulp waste	3	66.4631				
	OV90 Pulp waste	3	67.7878				
	VD60 Pulp waste	3		71.8575			
	OV50 Pulp waste	3			80.2784		
	VD70 Pulp waste	3			80.9524		
	OV80 Pulp waste	3			81.0931		
	VD80 Pulp waste	3			81.3030		
	OV70 Pulp waste	3			81.4198		
	VD90 Pulp waste	3			81.4782		
	OV60 Pulp waste	3				85.1357	
	FD Pulp waste	3					90.4653
	Sig.		.139	1.000	.231	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 3.000.

Table D15 Duncan table for effect of storage time on naringin of FC

NAR_FC

	Sample	N	Subset for alpha = 0.05					
			1	2	3	4	5	6
Duncan[a,b]	Week_8	2	26.1150					
	Week_11	3	28.5900	28.5900				
	Week_7	2	28.8300	28.8300				
	Week_9	2	29.3200	29.3200	29.3200			
	Week_4	2		31.3600	31.3600	31.3600		
	Week_12	2		31.5050	31.5050	31.5050		
	Week_5	2		31.6850	31.6850	31.6850		
	Week_3	2			32.6400	32.6400	32.6400	
	Week_10	2				33.1300	33.1300	
	Week_2	2				34.0450	34.0450	34.0450
	Week_1	3				34.1433	34.1433	34.1433
	Week_6	2					35.4550	35.4550
	Week_0	3						36.6900
	Sig.		.055	.072	.052	.105	.095	.108

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.167.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Table D16 Duncan table for effect of storage time on naringin of VC

NAR_VC

	Sample	N	Subset for alpha = 0.05				
			1	2	3	4	5
Duncan[a,b]	Week_10	3	46.6333				
	Week_9	2	47.6050	47.6050			
	Week_11	3	47.6633	47.6633			
	Week_4	3	51.6300	51.6300	51.6300		
	Week_12	3	52.4667	52.4667	52.4667		
	Week_5	3	52.5667	52.5667	52.5667		
	Week_7	3	52.9667	52.9667	52.9667		
	Week_8	2		54.3250	54.3250		
	Week_2	3			54.9400	54.9400	
	Week_6	3			56.8933	56.8933	
	Week_3	2			57.4200	57.4200	
	Week_1	3				61.2267	
	Week_0	3					67.9533
	Sig.		.075	.059	.104	.064	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.690.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Table D17 Duncan table for effect of storage time on DPPH for FC

DPPH_FC

	Sample	N	Subset for alpha = 0.05									
			1	2	3	4	5	6	7	8	9	10
Duncan[a]	Week_9	3	3074.6267									
	Week_5	3	3139.1967	3139.1967								
	Week_8	3	3191.5367	3191.5367	3191.5367							
	Week_7	3	3250.1567	3250.1567	3250.1567	3250.1567						
	Week_13	3		3364.8543	3364.8543	3364.8543	3364.8543					
	Week_12	3			3405.1200	3405.1200	3405.1200					
	Week_4	3			3420.7100	3420.7100	3420.7100					
	Week_11	3				3475.0167	3475.0167	3475.0167				
	Week_6	3					3503.8133	3503.8133	3503.8133			
	Week_10	3						3665.1333	3665.1333	3665.1333		
	Week_3	3							3715.6233	3715.6233	3715.6233	
	Week_2	3								3770.4100	3770.4100	
	Week_1	3									3931.6800	
	Week_0	3										4613.0900
	Sig.		.140	.059	.061	.066	.251	.099	.067	.356	.062	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 3.000.

Table D17 Duncan table for effect of storage time on DPPH for VC

	Sample	N	Subset for alpha = 0.05					
			1	2	3	4	5	6
Duncan[a]	Week_9	3	3207.3600					
	Week_8	3	3259.5333					
	Week_7	3	3448.6333					
	Week_12	3	3478.9833					
	Week_10	3	3482.5600					
	Week_4	3		3888.0167				
	Week_11	3		3944.6033				
	Week_13	3		4149.8400	4149.8400			
	Week_6	3			4228.7633			
	Week_5	3			4370.2500	4370.2500		
	Week_1	3				4603.3900	4603.3900	
	Week_3	3					4778.7600	
	Week_2	3					4846.8067	
	Week_0	3						5775.9900
	Sig.		.061	.061	.112	.078	.080	1.000

Table D18 Statistical analysis of pearson correlation during storage

		NAR	DPPH	TPC	FRAP
NAR	Pearson Correlation				
	Sig. (2-tailed)				
DPPH	Pearson Correlation	.622**			
	Sig. (2-tailed)	.000			
TPC	Pearson Correlation	.946**	.566**		
	Sig. (2-tailed)	.000	.000		
FRAP	Pearson Correlation	.910**	.623**	.930**	1
	Sig. (2-tailed)	.000	.000	.000	

**. Correlation is significant at the 0.01 level (2-tailed).
b. Listwise N=59

E. Condition of sample during drying

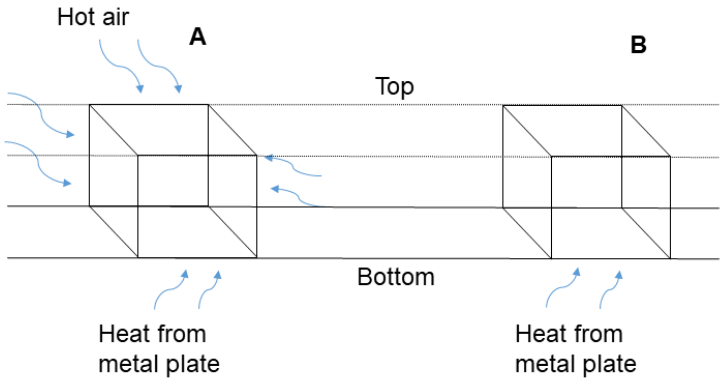

Figure E1 Condition of cross section of pomelo albedo slices during A) conventional drying and B) vacuum drying

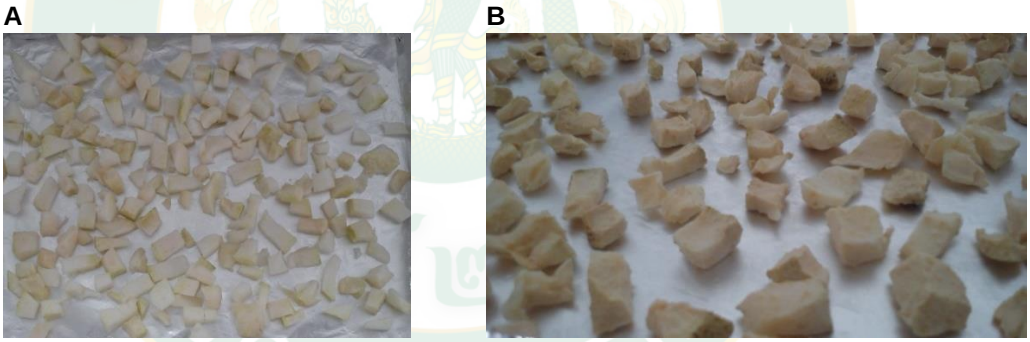

Figure E2 Fresh and Dried albedo

E. Effects of drying methods and temperature on color (Hue, Chroma and Browning Index (BI)) of pomelo residues

Table F1 Effects of different drying treatment and temperature on color of pomelo residues

Pomelo residues	Treatment	Temp.	hue	Chroma	BI
Flavedo	FD		84.88 ± 0.48^{a}	37.02 ± 0.09^{b}	85.07 ± 0.70^{j}
	CD	50	84.90 ± 1.51^{a}	37.19 ± 0.73^{b}	96.64 ± 2.58^{i}
		60	83.74 ± 0.47^{b}	34.84 ± 0.14^{c}	99.79 ± 2.71^{h}
		70	81.35 ± 0.53^{cd}	38.98 ± 0.74^{a}	115.11 ± 2.83^{fg}
		80	81.71 ± 0.58^{c}	39.75 ± 0.61^{a}	112.28 ± 0.78^{g}
		90	67.86 ± 0.48^{f}	37.25 ± 0.85^{b}	157.21 ± 2.16^{a}
	VD	50	80.54 ± 0.07^{d}	27.37 ± 0.14^{e}	117.59 ± 0.22^{ef}
		60	80.56 ± 0.11^{d}	27.64 ± 0.06^{e}	124.35 ± 0.76^{d}
		70	81.44 ± 0.33^{cd}	29.77 ± 0.33^{d}	118.70 ± 2.32^{e}
		80	81.20 ± 0.19^{cd}	29.85 ± 0.25^{d}	128.96 ± 0.83^{c}
		90	79.50 ± 0.29^{e}	30.00 ± 0.25^{d}	137.91 ± 0.43^{b}
Albedo	FD		82.21 ± 0.37^{a}	24.86 ± 0.04^{i}	45.83 ± 0.47^{h}
	CD	50	73.33 ± 1.09^{f}	31.95 ± 0.25^{d}	65.06 ± 0.89^{f}
		60	76.53 ± 0.83^{bcd}	35.62 ± 0.24^{c}	78.00 ± 1.09^{e}
		70	75.79 ± 0.33^{bcde}	31.22 ± 0.18^{e}	59.20 ± 0.87^{g}
		80	75.36 ± 1.03^{e}	38.08 ± 0.98^{b}	76.98 ± 1.75^{e}
		90	66.07 ± 0.79^{g}	41.76 ± 0.11^{a}	140.76 ± 1.19^{a}
	VD	50	77.09 ± 0.08^{b}	27.73 ± 0.11^{h}	92.81 ± 1.10^{d}
		60	76.92 ± 0.18^{bc}	28.26 ± 0.35^{h}	91.05 ± 1.04^{d}
		70	76.96 ± 0.26^{b}	29.08 ± 0.29^{g}	92.40 ± 1.32^{d}
		80	76.21 ± 0.21b^{cde}	30.61 ± 0.10^{f}	99.17 ± 0.67^{c}
		90	75.62 ± 0.51^{de}	31.79 ± 0.26^{de}	110.25 ± 0.58^{b}

Table F1 (Continued**)**

Lamella	FD		77.95 ± 0.46^{ab}	25.70 ± 0.06^{d}	49.97 ± 0.32^{g}
	CD	50	72.71 ± 1.27^{g}	29.82 ± 0.37^{de}	61.13 ± 1.23^{f}
		60	74.98 ± 0.71^{ef}	34.86 ± 0.20^{b}	76.52 ± 0.67^{d}
		70	75.54 ± 0.55^{de}	32.43 ± 0.9^{c}	66.77 ± 3.14^{e}
		80	74.17 ± 0.10^{f}	35.31 ± 1.41^{b}	76.21 ± 4.08^{d}
		90	68.25 ± 0.17^{h}	39.30 ± 0.54^{a}	118.77 ± 3.48^{a}
	VD	50	77.59 ± 0.86^{bc}	27.92 ±0.50^{f}	100.57 ± 3.42^{b}
		60	76.39± 0.81^{d}	29.15 ± 0.34^{e}	98.58 ± 4.55^{b}
		70	78.62 ± 0.25^{ab}	29.54 ± 0.19^{de}	91.51± 0.79^{c}
		80	79.02 ± 0.27^{a}	29.74 ± 0.08^{de}	90.09 ± 1.19^{c}
		90	76.58± 0.29^{dc}	30.74 ± 1.37^{d}	103.81± 5.79^{b}
Pulp waste	FD		86.55±0.53^{a}	28.83±0.47^{e}	60.99±1.73^{f}
	CD	50	65.36±0.54^{d}	35.35±1.29^{c}	139.97±3.23^{a}
		60	67.67±0.71^{cd}	37.30±0.56^{b}	121.33±1.69^{bc}
		70	70.05±1.38^{bc}	39.84±0.76^{a}	107.22±5.85^{d}
		80	72.85±0.39^{b}	35.72±0.33^{c}	92.06±1.90^{e}
		90	64.26±6.64^{d}	28.80±1.20^{e}	143.35±2.59^{a}
	VD	50	86.84±0.21^{a}	29.03±0.90^{e}	108.66±2.34^{d}
		60	86.50±0.07^{a}	31.76±0.12^{d}	117.26±2.30^{c}
		70	85.95±0.07^{a}	32.52±0.36^{d}	123.73±1.45^{b}
		80	85.84±0.15^{a}	32.90±0.50^{d}	124.54±3.47^{b}
		90	85.01±0.34^{a}	31.87±0.09^{d}	125.87±2.41^{b}

Values are mean ± SD, significant different ($p < 0.05$) of data were represent with the small letter (a,b,c) within the column.

FD: Freeze drying; CD: conventional drying; VD: Vacuum drying.

F. Effects of drying methods and drying temperature on moisture content, total phenolic content and DPPH radical scavenging using factorial design of albedo

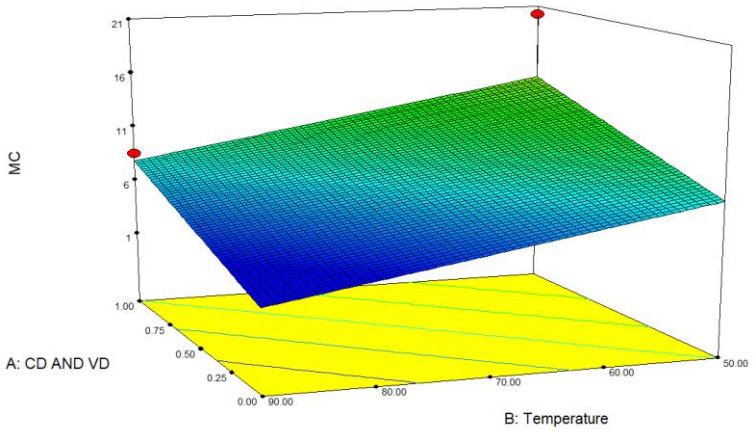

Figure G1 Effect of drying methods (A) and temperature (B) on moisture content

Table G1 Analysis of variance (ANOVA) for the effects of drying methods and temperature on moisture content

Source	Coefficient estimates	Sum of Squares	Degree of freedom	Mean Square	F-Value	p-value, Prob > F
Intercept	4.82	-	1			
Model	-	145.38	2	72.69	7.57	0.0178*
CD,VD	6.19	95.90	1	95.90	9.99	0.0159*
Temperature	-3.15	49.48	1	49.48	5.15	0.0574
Residual		67.19		9.60		
Cor Total		212.57	9			
Lack of fit						
R^2	0.6839					

*Significant (<0.0500)

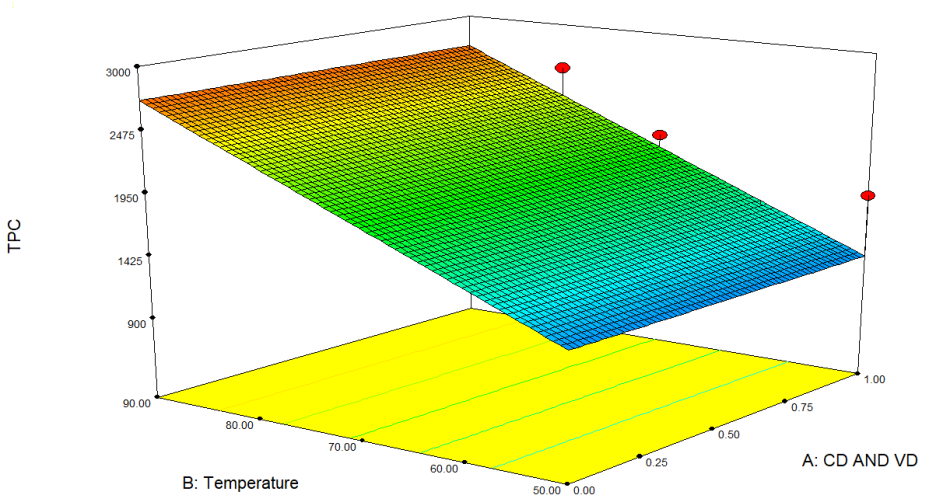

Figure G2 Effect of drying methods (A) and temperature (B) on TPC

Table G2 Analysis of variance (ANOVA) for the effects of drying methods and temperature on TPC

Source	Coefficient estimates	Sum of Squares	Degree of freedom	Mean Square	F-Value	p-value, Prob > F
Intercept	1994.06	-	1			
Model	-	2.683E+006	2	1.341E+006	9.64	0.0098*
A-CD,VD	0.61	0.93	1	0.93	6.692E-006	0.9980
B-Temperature	732.52	2.683E+006	1	2.683E+006	19.27	0.0032*
Residual		9.746E+005	7	1.392E+005		
Cor Total		3.657E+006	9			
Lack of fit						
R^2	0.7335					

* significant (<0.0500)

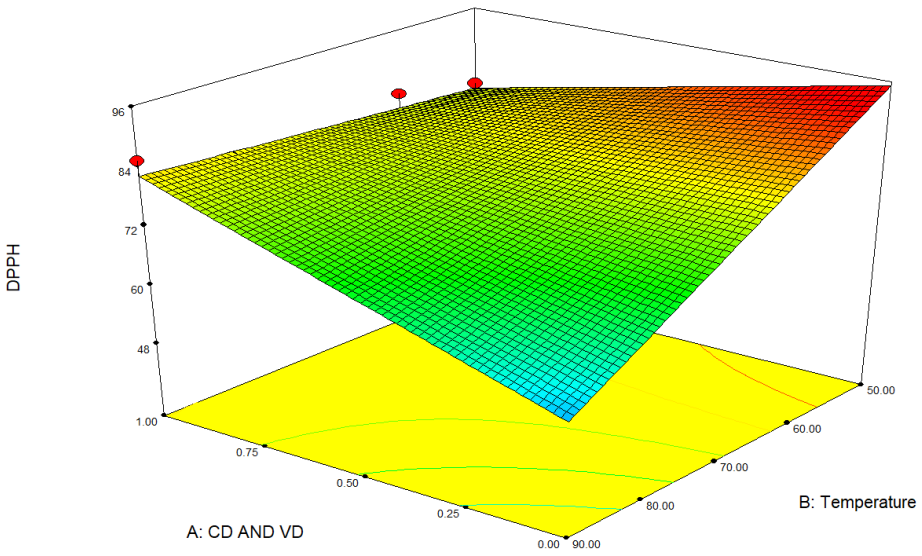

Figure G3. Effect of drying methods (A) and temperature (B) on DPPH

Table G3 Analysis of variance (ANOVA) for the effects of drying methods and temperature on DPPH.

Source	Coefficient estimates	Sum of Squares	Degree of freedom	Mean Square	F-Value	p-value, Prob > F
Intercept	75.46	-	1			
Model	-	1054.17	3	351.39	6.02	0.0306*
A-CD,VD	5.11	65.20	1	65.20	1.12	0.3314
B-Temperature	-19.80	980.47	1	980.47	16.79	0.0064*
AB	21.65	585.75	1	585.75	10.03	0.0194*
Residual		350.46	6	58.41		
Cor Total		1404.63	9			
Lack of fit						
R^2	0.7505					

*Significant (<0.0500)

BIOADATA OF STUDENT

Nur Farhana Abd Rahman was born in Kuala Lumpur on 3rd March 1988. She attended primary school at Sekolah Kebangsaan Sultan Hishamuddin Alam Shah, KL and had her secondary education at SMKA Maahad Hamidiah, Kajang. In 2006, she enrolled in Johor Matriculation College (JMC) at Tangkak, Johor. In 2007, she was offered to continue her study at Universiti Teknologi MARA (UiTM), Shah Alam as a bachelor student of Food Science and Technology (Minor : Halal Islamic Food Law) (Hons.) in Faculty of Applied Science. In 2010, she had joined an exhibition of Invention, Innovation and Design (IID), Special Edition and she had been awarded with bronze medal (Innovation Categories). In 2011, she had been selected to undergo practical training at Bogor Agricultural University, Dramaga, Bogor, Indonesia around 5 month under Malaysia-Indonesia-Thailand (MIT) mobility program which has been organized by Ministry of Higher Education (MOHE), Malaysia. After graduated in 2011, she had experience worked at Segi Food Services Sdn Bhd as QC and Food Technologist. In February 2012 until May 2015, she completed in master program with Halal Product Development at Halal Product Research Institute, Universiti of Putra Malaysia. In 2015, she earned scholarship from Graduate Research Funding (GRF) from UPM in the early semester and for the consequence semester she was offered scholarship from Ministry of Higher Education (MOHE), Malaysia for continuing study in Dual Degree PhD Programmed between UPM, Malaysia and Maejo University, Thailand under International Collaborative Programme.

LIST OF PUBLICATIONS

Rahman, N. F. A., Shamsudin, R., Ismail, A., & Shah, N. N. A. K. (2016). Effects of post-drying methods on pomelo fruit peels. *Food Science and Biotechnology*, *25*(S1), 85–90. https://doi.org/10.1007/s10068-016-0102-y (Status: Published)

Rahman, N. F. A., Shamsudin, R., Ismail, A., Shah, N. N. A. K. & Varith J. (2018). Effect of drying methods on antioxidant properties of pomelo peels. *Innovative Food Science and Emerging Technologies*, 50, 217-225

Rahman, N. F. A., Shamsudin, R., Ismail, A., Shah, N. N. A. K. & Varith J. (2019). Effect of drying temperature on Malaysia pomelo (*Citrus grandis* (L.) Osbeck) pomace residue under vacuum condition. Pertanika Journal Science and Technology. 27 (S1): 57-66.

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Nur Farhana Abd Rahman, Rosnah Shamsudin, Amin Ismail, Nor Nadiah Abdul Karim Shah, Jaturapatr Varith.(2017) Influence of drying methods on the bioactive compound and antioxidant activity of pomelo residues. *The International Food Research Conference (IFRC 2017), Complex of the Deputy Vice Chancellor (Research and Innovation), Universiti Putra Malaysia,* 25-27 July 2017.

Nur Farhana Abd Rahman, Rosnah Shamsudin, Amin Ismail, Nor Nadiah Abdul Karim Shah, Jaturapatr Varith.(2018). Nutrisi Kulit Limau Bali. *Malaysian Society Of Agricultural Engineers (MSAE) Conference, Faculty of Engineering,Universiti Putra Malaysia.*7-8 Februari 2018.

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Nur Farhana Abd Rahman, Rosnah Shamsudin, Amin Ismail, Nor Nadiah Abdul Karim Shah, Jaturapatr Varith. (2018). Physicochemical Properties of Dried Pomelo Wastes (*Citrus Grandis* (L.) Osbeck) Byproducts. The 6th Symposium on Applied Engineering and Sciences 2018 (SAES2018), Kyushu Institute of Technology, Fukuoka, Japan, 15 - 16 Dec 2018.

Nur Farhana Abd Rahman, Rosnah Shamsudin, Amin Ismail, Nor Nadiah Abdul Karim Shah, Jaturapatr Varith. (2018). Physicochemical Properties of Dried Pomelo (*Citrus Grandis* (L.) Osbeck) Byproducts. Konvensyen Kebangsaan Kejuruteraan Pertanian Dan Makanan 2019, Wisma Tani, Kementerian Pertanian Malaysia, Putrajaya, 21 Mac 2019

Articles- to be submitted.

Rahman, N. F. A., Shamsudin, R., Varith J., Ismail, A. & Shah, N. N. A. K. Effect of Drying Methods on Nutritional Composition of Malaysia's Pomelo Peels (Status: Manuscript to be submitted to International Journal of Food Science and Nutrition).

Rahman, N. F. A., Shamsudin, R., Varith J., Ismail, A. & Shah, N. N. A. K. Influence of storage on naringin compound of Tambun White pomelo's albedo. (Status: Manuscript to be submitted to Journal of Food Engineering).

Rahman, N. F. A., Shamsudin, R., Varith J., Ismail, A. & Shah, N. N. A. K. Influence of conventional and vacuum drying on quality of pomelo (*Citrus grandis* (L)

Osbeck) byproduct. (Status: Manuscript to be submitted to Journal of Food Engineering).

CURRICULUM VITAE

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