Taylor & Francis
Taylor & Francis Group

Food Reviews International

ISSN: 8755-9129 (Print) 1525-6103 (Online) Journal homepage: http://www.tandfonline.com/loi/lfri20

Pretreatment of citrus by-products affects polyphenol recovery: a review

Konstantinos Papoutsis, Quan V. Vuong, John B. Golding, Joaquín H. Hasperué, Penta Pristijono, Michael C. Bowyer, Christopher J. Scarlett & Costas E. Stathopoulos

FOOD REVIEWS INTERNATIONAL
https://doi.org/10.1080/87559129.2018.1438471

Pretreatment of citrus by-products affects polyphenol recovery: a review

Konstantinos Papoutsis [a], Quan V. Vuong[a], John B. Golding[a,b], Joaquín H. Hasperué[d], Penta Pristijono[a], Michael C. Bowyer[a], Christopher J. Scarlett[a], and Costas E. Stathopoulos[c]

[a]School of Environmental and Life Sciences, The University of Newcastle, Ourimbah, NSW, Australia; [b]NSW Department of Primary Industries, Ourimbah Campus, Ourimbah, Australia; [c]Division of Food and Drink School of Science, Engineering and Technology, University of Abertay, Dundee, UK; [d]Center for Research and Development in Food Cryotechnology (CIDCA, National University of La Plata-CONICET), La Plata, Buenos Aires, Argentina

ABSTRACT
A large amount of citrus waste is generated annually. This waste is of great economic worth, since it contains high levels of polyphenols, which have attracted scientific interest due to their potent antimicrobial and antiradical activities. Pretreatment is a crucial step that precedes the extraction process and influences the yields and quality of polyphenols. This review emphasizes the effect of different drying processes, such as freeze drying, hot-air drying, vacuum drying, microwave drying, infrared drying, and high-speed drying, on the polyphenol retention in citrus by-products. Further treatments of the dried citrus by-products for assisting the liberation of bound polyphenols are also provided and comprehensively discussed.

KEYWORDS
Citrus pomace; drying techniques; lyophilization; polyphenol liberation; treatment of dried pomace; valorization

Introduction

Citrus is one of the most economically important crops with a worldwide production exceeding 121 × 10^6 tons. [1] Citrus fruits are usually processed by the industry for the production of juice. [2,3] During juice production, a large amount of citrus by-products are generated, mainly consisting of peel and seed residues. This material has recently attracted scientific interest since several studies have pointed out that citrus by-product (also known as pomace) is a good source of polyphenols, which have been linked to antimicrobial [4], antifungal [5], anticancer [6], and antioxidant activities. [7,8] It is known that polyphenols can be degraded by exposure to light, oxygen, and high temperature. [9,10] Therefore, citrus by-products should be appropriately handled for retaining high polyphenol content.

Phenolic compounds are the most abundant secondary metabolites synthesized by plants as a response to external stresses, such as ultraviolet (UV) irradiation, wounding, pathogen attack, or during plant maturation. [11,12] Flavonoids and phenolic acids are the main classes of polyphenols found in citrus by-products. [13] More than 60 flavonoid compounds have been identified in citrus and sub-classified as flavanones, flavones, and flavonols [14] (Table 1). Citrus flavonoids are present in glycoside (C- or O-glycosides) or

CONTACT Konstantinos Papoutsis Konstantinos.Papoutsis@uon.edu.au Nutrition Food & Health Research Group, School of Environmental and Life Sciences, University of Newcastle, Brush Rd, Ourimbah, NSW 2258, Australia.
Color versions of one or more of the figures in the article can be found online at www.tandfonline.com/lfri.

Table 1. Flavonoid contents of the peels of different citrus species.

Flavonoids	Citrus species	Quantity	Drying technique applied	Refs
Flavanones				
Hesperidin	Orange	66.10 mg/g d.w.[1]	Freeze drying	[78,79,78,80,81,81]
	Orange	30.17-39.09 mg/g d.w.[2]	Freeze drying	
	Grapefruit	1.67-3.87 mg/g d.w.[1]	Freeze drying	
	Mandarin	55.26 mg/g[3]	Vacuum drying (60 °C)	
	Mandarin	29.5 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
	C. tankan Hayata	23.4 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
Naringin	Grapefruit	4.04-5.91 mg/g d.w.[1]	Freeze drying	[78,82,83,83]
	Mandarin	19.49 mg/g f.w.[1]	Freeze drying	
	Lemon	6.06 mg/g d.w.[4]	Hot-air drying (40 °C)	
	Sour orange	10.97 mg/g d.w.[4]	Hot-air drying (40 °C)	
Neoeriocitrin	Lemon	6.12 mg/g d.w.[4]	Hot-air drying (40 °C)	[83,83,82,82]
	Sour orange	3.80 mg/g d.w.[4]	Hot-air drying (40 °C)	
	Orange	8.80 mg/g f.w.[1]	Freeze drying	
	Mandarin	34.65 mg/g f.w.[1]	Freeze drying	
Neohesperidin	Sour orange	6.62 mg/g d.w.[4]	Hot-air drying (40 °C)	[83,83,82,40]
	Lemon	4.37 mg/g d.w.[4]	Hot-air drying (40 °C)	
	Mandarin	7.09 mg/g f.w.[1]	Freeze drying	
	Calamondin	3.49 mg/g d.w.[4]	Freeze drying	
Narirutin	Sour orange	0.25 mg/g d.w.[4]	Hot-air drying (40 °C)	[83,82,79,82]
	Orange	16.52 mg/g f.w.[1]	Freeze drying	
	Orange	2.40-5.25 mg/g d.w.[2]	Freeze drying	
	Mandarin	13.54 mg/g f.w.[1]	Freeze drying	
Didymin	Orange	1.10-1.69 mg/g d.w.[2]	Freeze drying	[79,79]
	Mandarin	0.31-1.23 mg/g d.w.[2]	Freeze drying	
Flavonols				
Rutin	Lemon	0.29 mg/g d.w.[4]	Hot-air drying (below 50 °C)	[81,82,81,81]
	Orange	8.16 mg/g f.w.[1]	Freeze drying	
	Orange	0.23 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
	Mandarin	0.29 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
Quercetin	Mandarin	0.47 mg/g d.w.[4]	Hot-air drying (below 50 °C)	[81,81,81]
	Orange	0.14 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
	Lemon	0.21 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
Catechin	Mandarin	0.02 mg/g d.w.[3]	Sun drying	[16]
Kaempferol	Orange	0.32 mg/g d.w.[4]	Hot-air drying (below 50 °C)	[81,81,81]
	Mandarin	0.38 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
	Lemon	0.31 mg/g d.w.[4]	Hot-air drying (below 50 °C)	
Flavones				
Diosmin	Orange	4.55 mg/g f.w.[1]	Freeze drying	[82,79,82,79,84]
	Orange	0.49-0.53 mg/g d.w.[2]	Freeze drying	
	Mandarin	6.29 mg/g f.w.[1]	Freeze drying	
	Mandarin	1.02-1.40 mg/g d.w.[2]	Freeze drying	
	Lemon	3.30 mg/g d.w.[1]	Hot-air drying (50 °C)	
Tangeretin	Calamondin	0.07-1.95 mg/g d.w.[4]	Freeze drying	[40,79,79,85]
	Orange	0.16-0.33 mg/g d.w.[2]	Freeze drying	
	Mandarin	0.27-0.86 mg/g d.w.[2]	Freeze drying	
	Satsuma mandarin	0.16 mg/g d.w.[1]	Hot-air drying (45 °C)	
Nobiletin	Orange	0.41-0.65 mg/g d.w.[2]	Freeze drying	[79,79,85,40]
	Mandarin	0.45-0.61 mg/g d.w.[2]	Freeze drying	
	Satsuma mandarin	0.31 mg/g d.w.[1]	Hot-air drying (45 °C)	
	Calamondin	0.86-2.70 mg/g d.w.[4]	Freeze drying	

d.w.: dry weight; f.w.: fresh weight
[1]: Quantification by HPLC-Diode array detector (DAD)
[2]: Quantification by HPLC-Diode array detector (DAD) and HPLC-MS
[3]: Quantification by HPLC-Photodiode array (PDA) detector
[4]: Quantification by HPLC-UV detector

aglycone forms, with the glycoside being the dominant form. [13] Phenolic acids are phenols containing one carboxylic acid and divided into hydroxycinnamic and hydroxybenzoic acids. [15] Phenolic acids in plant tissues are present in free and bound forms, with the free forms being more active than the bound ones. [16] Table 2 illustrates the phenolic acid content of different citrus species peels.

Citrus by-products are exposed to undesirable environmental conditions (oxygen and high temperatures), which might lead to polyphenol degradation. Therefore, for obtaining high polyphenol extraction yields, apart from the selection of the most efficient extraction technique, the preservation of polyphenols on the citrus by-product should be considered. Citrus by-products are prone to degradation and spoilage due to their high moisture content. Usually, drying precedes extraction for moisture removal and polyphenol preservation. However, undesirable drying conditions may cause degradation of polyphenols, leading to lower extraction yields. A number of drying techniques have been employed for the dehydration of citrus by-products, such as freeze drying, hot-air drying, vacuum drying, sun drying, infrared drying, far-infrared drying, and high-speed drying, demanding different energy requirements and exerting various effects on the different classes of polyphenols. [9,10,17–19] For instance, drying at high temperatures may result in the liberation of the bound polyphenols facilitating greater recovery. However, at high temperatures, some polyphenols might be oxidized and converted to other compounds. [20] When the drying process is conducted under sunlight, a reduction in the retention of polyphenols may occur due to photo-oxidation. [21] The merits and demerits of each drying technique are summarized in Table 3 and are comprehensively discussed in the following sections.

As previously mentioned, polyphenols in citrus by-products are often linked to cell wall polymers. Recent studies have shown that the liberation of bound polyphenols from citrus by-products can be achieved by applying further treatments, such as heat (conventional or microwave ovens) or irradiation (electron-beam and UV-C irradiations) to the dried materials. [16,22–26] During the heat treatment, the ester and glucoside bonds can be broken down, resulting in the liberation of the bound polyphenols. At the same time, heat may lead to polyphenol transformation explaining why the content of some phenolic compounds increases, while the content of others decreases. [27] In case of irradiation, the exact mechanism has not yet been elucidated; however, it could be hypothesized, that the higher polyphenol yields might be due to the breakdown of the bound polyphenols caused by heating effects due to the transfer of energy of many photons. [28]

To date, several reviews have been published presenting and discussing the different extraction techniques that have been applied on citrus by-products for the recovery of polyphenols. [29–31] However, to the best of our knowledge, there is no review discussing the effect of different drying conditions on the recovery of polyphenols from citrus by-products. Therefore, this review aims to provide information about the effect of different drying techniques and conditions on the polyphenol recovery from citrus by-products. Moreover, additional steps that are used to enhance the extraction procedure for liberating and obtaining high-quality polyphenol extracts are comprehensively discussed.

Table 2. Phenolic acid contents of the peels of different citrus species.

Phenolic acids	Type of material	Quantity	Drying technique applied	Refs
Hydroxycinnamic acids				
Ferulic acid	Mandarin	2.39 mg/g d.w.[1]	Sun drying	[68,86,85,16,87,86,86,88,88,64]
	Mandarin	0.01 mg/g d.w.[2]	Freeze drying	
	Satsuma mandarin	2.76 mg/g d.w.[3]	Hot-air drying (45 °C)	
	Mandarin	0.85 mg/g d.w.[1]	Sun drying	
	Satsuma mandarin	0.95-1.42 mg/g d. w.[1]	Hot-air drying (40 °C)	
	Lemon	0.02 mg/g d.w.[2]	Freeze drying	
	Orange	0.02 mg/g d.w.[2]	Freeze drying	
	Orange	0.18 mg/g[2]	N.M.	
	Grapefruit	0.16 mg/g[2]	N.M.	
	Grapefruit	0.01-0.14 mg/g d. w.[1]	Hot-air drying (45 °C)	
p-Coumaric acid	Mandarin	0.83 mg/g d.w[1]	Sun drying	[68,86,85,16,87,86,86,88,88,64]
	Mandarin	0.05 mg/g d.w.[2]	Freeze drying	
	Satsuma mandarin	0.30 mg/g d.w.[3]	Hot-air drying (45 °C)	
	Mandarin	0.35 mg/g d.w.[1]	Sun drying	
	Satsuma mandarin	0.02-0.18 mg/g d. w.[1]	Hot-air drying (40 °C)	
	Lemon	0.04 mg/g d.w.[2]	Freeze drying	
	Orange	0.01 mg/g d.w.[2]	Freeze drying	
	Orange	0.08 mg/g[2]	N.M.	
	Grapefruit	0.02 mg/g[2]	N.M.	
	Grapefruit	0.01-0.07 mg/g d. w.[1]	Hot-air drying (45 °C)	
Caffeic acid	Mandarin	0.01 mg/g d.w.[1]	Freeze drying	[86,85,87,86,86,88,88,64]
	Satsuma mandarin	0.14 mg/g d.w.[3]	Hot-air drying (45 °C)	
	Satsuma mandarin	0.04-0.06 mg/g d. w.[1]	Hot-air drying (40 °C)	
	Lemon	0.02 mg/g d.w.[2]	Freeze drying	
	Orange	0.02 mg/g d.w.[2]	Freeze drying	
	Orange	0.01 mg/g[2]	N.M.	
	Grapefruit	0.01 mg/g[2]	N.M.	
	Grapefruit	0.001-0.01 mg/g d. w.[1]	Hot-air drying (45 °C)	
Sinapic acid	Mandarin	0.02 mg/g d.w.[1]	Freeze drying	[86,85,87,86,86,88,88,64]
	Satsuma mandarin	0.19 mg/g d.w.[3]	Hot-air drying (45 °C)	
	Satsuma mandarin	0.12-0.16 mg/g d. w.[1]	Hot-air drying (40 °C)	
	Lemon	0.07 mg/g d.w.[2]	Freeze drying	
	Orange	0.01 mg/g d.w.[2]	Freeze drying	
	Orange	0.10 mg/g[2]	N.M.	
	Grapefruit	0.03 mg/g[2]	N.M.	
	Grapefruit	0.001-0.05 mg/g d. w.[1]	Hot-air drying (45 °C)	
Chlorogenic acid	Grapefruit	0.02-0.13 mg/g d. w.[1]	Hot-air drying (45 °C)	[64,86,86,86]
	Lemon	0.09 mg/g d.w.[2]	Freeze drying	
	Orange	0.02 mg/g d.w.[2]	Freeze drying	
	Mandarin	0.04 mg/g d.w.[2]	Freeze drying	
Hydroxybenzoic acids				
Vanillic acid	Mandarin	0.32 mg/g d.w.[1]	Sun drying	[68,85,16,87,64]

(*Continued*)

Table 2. (Continued).

	Satsuma mandarin	0.05 mg/g d.w.[3]	Hot-air drying (45 °C)	
	Mandarin	0.39 mg/g d.w.[1]	Sun drying	
	Satsuma mandarin	0.03 mg/g d.w.[1]	Hot-air drying (40 °C)	
	Grapefruit	0.001-0.16 mg/g d.w.[1]	Hot-air drying (45 °C)	
p-Hydroxybenzoic acid	Mandarin	0.07 mg/g d.w.[1]	Sun drying	[68,85,16,87,64]
	Satsuma mandarin	0.06 mg/g d.w.[3]	Hot-air drying (45 °C)	
	Mandarin	0.30 mg/g d.w.[1]	Sun drying	
	Satsuma mandarin	0.03 mg/g d.w.[1]	Hot-air drying (40 °C)	
	Grapefruit	0.002-0.01 mg/g d.w.[1]	Hot-air drying (45 °C)	
Gallic acid	Mandarin	0.18 mg/g d.w.[1]	Sun drying	[68,16]
	Mandarin	0.18 mg/g d.w.[1]	Sun drying	

d.w.: dry weight.; N.M.: not mentioned
[1]: Quantification by HPLC-Photodiode array (PDA) detector
[2]: Quantification by HPLC-UV detector
[3]: Quantification by HPLC-Diode array detector (DAD)

Impact of drying techniques on citrus by-product polyphenols

Drying is a crucial but not mandatory step that precedes the extraction process (Fig. 1). Citrus by-products contain a significant amount of moisture which promotes microbial spoilage and chemical alterations. [32–34] Even though citrus by-products could be stored in a freezer as fresh, dehydration is recommended for several reasons. After dehydration, (i) the activity of enzymes that are responsible for the degradation of polyphenols, such as polyphenol oxidase (PPO) or peroxidase, is significantly reduced, therefore, higher polyphenol yields are obtained [33,35] (Fig. 2), (ii) citrus wastes are not vulnerable to pathogen spoilage [36], and (iii) the packaging volume is significantly lower, hence less storage place is required and the transportation cost is reduced.

A number of studies have indicated that undesirable drying conditions may negatively affect the extraction yields of polyphenols from citrus by-products. [9,10,37] Several parameters should be considered for the selection of the drying technique, including energy consumption, retention of polyphenols in the dried material, as well as its effect on the physical properties of the dried product. The application and effects of different techniques on the retention of polyphenols are discussed in the following sections. Table 4 illustrates the studies that have been conducted to evaluate the effect of different drying techniques on the retention of polyphenols in citrus by-products.

Freeze drying (lyophilization)

Freeze drying or lyophilization is a drying technique operating at low temperature and under vacuum. [35,38] Freeze drying has been widely used for drying citrus by-products [7,39,40] since, unlike other techniques, it prevents the discoloration of the dried material, it prohibits the degradation of heat or oxygen sensitive bioactive compounds, and it effectively removes the moisture from the materials. Hence, it promotes the storability of the product by minimizing

Table 3. Merits and demerits of the different drying processes that have been applied on citrus by-products.

Drying technique	Merits	Demerits	Refs
Freeze drying	• Promotes the storability of the product by minimizing pathogen and enzyme activity. • Prevents the polyphenol enzymatic degradation. • Prevents the degradation of the heat sensitive polyphenols. • The change of the citrus by-product color is negligible.	• Higher energy consumption and cost requirements compared with hot-air drying, vacuum drying, microwave drying, high-speed drying and infrared drying. • Requires longer drying times than hot-air drying, vacuum drying, microwave drying, high-speed drying, and infrared drying.	[10,18,19,45]
Hot-air drying	• Less energy consumption and cost requirements than freeze drying. • Requires shorter drying times than freeze drying and sun drying. • Promotes the liberation of some bound polyphenols.	• Brown color is developed as the temperature increases. • A degradation of the heat and oxygen sensitive polyphenols may occur.	[9,10,37,39,43,44,46]
Vacuum drying	• Less energy consumption requirements than freeze drying. • Lower processing temperatures compared to hot-air drying. • Promotes the liberation of some bound polyphenols. • Prevents the polyphenol enzymatic degradation.	• Brown color is developed as the temperature increases. • A degradation of the heat sensitive polyphenols may occur.	[10,55,56,89]
Sun drying	• Less energy consumption and cost requirements compared with the other drying techniques.	• Depends on environmental conditions (humidity, temperature, air velocity, and solar intensity). • Requires longer drying times than other drying techniques, including freeze drying. • A polyphenol enzymatic degradation may occur. • Microbial contamination of the dried material may occur.	[9,34,68]
Microwave drying	• Less energy consumption requirements than freeze drying, hot-air drying, vacuum drying, sun drying, infrared drying, and high-speed drying. • Requires shorter drying times compared to freeze drying, hot-air drying, vacuum drying, infrared drying, and high-speed drying. • Promotes the liberation of the bound polyphenols.	• Brown color is developed as the power and processing time increase. • A polyphenol enzymatic degradation may occur. • A degradation of the heat sensitive polyphenols may occur.	[9,18,19,42,60,89,90]
High-speed drying	• Requires shorter drying times than freeze drying, hot-air drying, vacuum drying, sun drying, and infrared drying. • Results in high retention of polyphenols in the dried material.	• The cost of equipment could be higher than other drying techniques, such as hot-air drying, vacuum drying, or sun drying.	[9,18,19,60,89,90]
Infrared drying	• Requires shorter drying time than freeze drying and sun drying.	• Results in lower retention of total polyphenols compared to freeze drying.	[9,18]

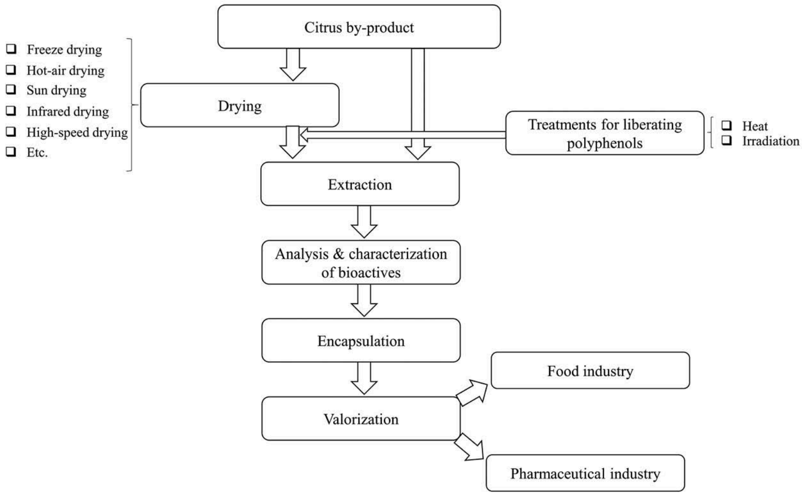

Figure 1. Steps for the valorization of polyphenols from citrus wastes.

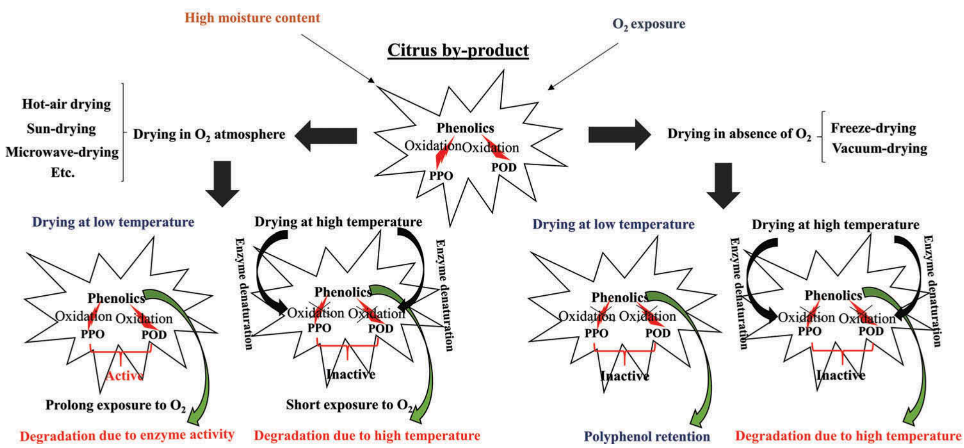

Figure 2. Effect of different drying conditions on the enzymatic degradation of polyphenols.

enzyme and pathogen activity. [29,41] However, it requires longer drying times (varying from 12 to 72 hours) and higher energy consumption than other drying techniques. [9,10,42,43]

According to the literature, freeze drying has different effects on the retention and recovery of different classes of polyphenols being found in citrus by-products. For instance, freeze drying could be effectively applied on citrus by-products for the retention of neoeriocitrin, neodiosmin, hesperidin, nobiletin, caffeic acid, and neohesperidin. [9,10,44,45] However, some contradictory results related to the effect of freeze drying on the retention of total phenolic content (TPC), antioxidants and phenolic acids on the dried citrus by-products have been noticed. [9,10,39,45] Sun et al. [9] indicated that freeze drying is a more effective technique for maintaining TPC, phenolic acids, and antioxidants in the peels of four citrus species comparing with hot-air drying (60°C) and sun drying. Similar results were reported by Assefa and Keum [42], who found higher retention of TPC and flavonoids in the citrus peels dried by freeze drying than those dried by

Table 4. Drying techniques applied on citrus by-products.

Drying treatments			
Citrus species	Drying procedures	Comments	Refs
Naranjita (*Citrus mitis* B.) by-products	• Hot-air drying (50–84°C)	• The drying process increased both TPC and antioxidant capacity. • Higher retention of TPC was observed in the dried by-products compared to the fresh ones. • The highest content of TPC can be obtained with the combination of high temperatures (>74°C) and low air velocity (<0.6 m/s).	[46]
Lemon (*Citrus limon*) by-products	• Freeze drying • Hot-air drying (70, 90, and 110°C) • Vacuum drying (70, 90, and 110°C)	• The TPC and antioxidant capacity were higher in the lemon by-products dried by hot-air or under vacuum than those dried by freeze drying. • The highest recovery of flavonoids was recorded in the by-products dried under vacuum at 70 and 90°C. • By-products dried under vacuum at 70°C had the highest rutin and *p*-coumaric acid content. • The highest recovery of gallic acid was recorded in the by-products dried by hot-air at 110°C.	[10]
Yuzu (*Citrus junos* Sieb ex Tanaka) peels	• Microwave drying • Hot-air drying (50°C) • Freeze drying • Air drying (25°C)	• All the drying methods resulted in higher values of TPC, flavonoid content, and antioxidant capacity compared to those obtained from the fresh samples. • Hot-air, microwave, and air drying resulted in the reduction of the TPC, flavonoid content, and antioxidant capacity compared to freeze drying. • Peels dried by hot-air drying had the same TPC, flavonoid content and antioxidant capacity with those dried by microwave drying. • Freeze drying resulted in the highest retention of TPC, flavonoid content, and antioxidants.	[42]
Lemon (*Citrus limon* v. lunari) peels	• Combined osmotic hot-air drying dehydration	• Significant loss of TPC (70–80%) was recorded during osmotic dehydration. • Osmotic dehydration process had protective effect against TPC loss during hot-air drying.	[54]
Lemon (*Citrus limon*) fruits	• Hot-air drying (45°C) • Freeze drying	• Sample dehydration provided extracts with higher amounts of flavonoids than fresh samples. • The flavonol content was higher in the extracts obtained from the hot-air dried samples compared to those dried by freeze drying. • The flavanone and flavone contents were higher in the extracts obtained from the freeze-dried samples compared to those dried by hot-air drying.	[41]

(*Continued*)

Table 4. (Continued).

Drying treatments			
Citrus species	Drying procedures	Comments	Refs
Lemon (*Citrus limon* cv. lunari) peels	• Hot-air drying (40, 50, and 60°C)	• Drying temperature had a significant effect on the TPC degradation rates. • Hot-air drying led to a significant reduction in the TPC and flavonoid content. • A first-order equation described the degradation of TPC and flavonoid content.	[37]
Oranges (*Citrus sinensis*) peels	• Hot-air drying (40, 60, and 80°C) • Freeze drying	• The highest content of major individual flavonoids and antioxidant activity of orange peel achieved at 80°C. • A rapid degradation of total and individual compounds occurred during the first hour of drying.	[53]
Kumquat (*Citrus japonica* var. margarita) fruits	• Hot-air drying (110, 130, and 150°C) followed by freeze drying	• When fruits were dried at 110 and 130°C, the TPC, antioxidant activity, and flavonoid content increased. • All of the flavonoids dramatically decreased after drying at 150°C. • The TPC and antioxidant capacity were higher in hot-air dried samples compared to the lyophilized.	[43]
Different citrus species	• Sun drying • Hot-air drying (60°C) • Freeze drying	• Freeze drying resulted in extracts with the highest phenolic acid content and antioxidant activity among the four citrus species. • The TPC of all citrus fruits was reduced after hot-air drying at 60°C. • Both hot-air and sun drying caused some loss of phenolic acids.	[9]
Calamondin (*Citrus mitis Blanco*) peels	• Hot-air drying (70, 85, 100, and 150°C) followed by freeze drying	• The peels dried at 150°C had the highest TPC and flavonoid content compared to the other treatments and fresh materials. • The heating enhanced the recovery of some phenolic compounds i.e., naringin, tangeretin, ferulic acid, *p*-coumaric acid, and gallic acid. • Some phenolic compounds, such as 3′,5′-di-C-β-glucopyranosylphloretin (DGPP), hesperidin and caffeic acid, were degraded during heating.	[45]
Orange (*Citrus sinensis*. v. Thompson); lemon (*Citrus limon*. v. lunari); mandarin (*Citrus reticulata*. v. Tangerine) peels	• Microwave drying (100–600 W)	• Microwave power significantly affected the retention of TPC in the different citrus species. • As the microwave power increased from 300 to 600 W the TPC of the microwave dried mandarin and orange peels increased. • Microwave power higher than 450 W resulted in the decrease of TPC of the microwave dried lemon peels.	[59]

(*Continued*)

Table 4. (Continued).

Drying treatments			
Citrus species	Drying procedures	Comments	Refs
Orange (*Citrus sinensis*) peels	• Microwave drying (100–850 W)	• Microwave power significantly affected the retention of TPC of the dried orange peels. • Drying power of 450 W was found to result in high amount of TPC retention.	[60]
Orange (*Citrus sinensis* L. Osbeck) peels	• Hot-air drying (50, 60, 70, 80, 90, and 100°C), • Freeze drying	• Lower TPC and flavonoid contents were recorder in the peels dried at lower temperatures (50 and 60°C), than to those dried at higher (70, 80, 90, and 100°C). • Peels dried at temperatures higher than 70°C had higher TPC compared to those dried by freeze drying. • Caffeic acid, *p*-coumaric acid, naringin, neohesperidin, kaempferol, and rutin contents were higher in the peels dried at 100°C, compared to those dried at lower temperatures or freeze dried.	[39]
Orange (*Citrus sinensis*) peels	• Infrared drying	• The TPC was higher in the peels dried at higher infrared drying temperatures. • The infrared drying peels retained lower TPC compared to the fresh ones.	[17]
Citrus by-products	• Far-infrared drying	• Far-infrared drying is effective for retaining specific phenolic compounds in citrus by-products. • The TPC of the extracts obtained from the far-infrared dried by-products was lower than those dried by freeze drying.	[18]
Citrus by-products	• High-speed drying • Freeze drying	• High-speed dried extracts showed high amount of polymethoxylated flavones (heptamethoxyflavone and nobiletin) and flavanones (hesperidin and narirutin). • The TPC and flavonoid content of the extracts obtained from the high-speed dried peels were close to those obtained by the freeze-dried ones.	[19]

hot-air drying (50°C), and those dried at ambient temperature. On the other hand, Lou et al. [45] indicated higher recovery of TPC and flavonoids from the immature calamondin peels after hot-air drying at 150°C compared to the freeze-dried ones. Chen et al. [39] found that orange peels dried at temperatures higher than 70°C had higher TPC compared to those dried by freeze drying. The different results could be attributed to the different drying operating conditions used in each study. In general, freeze drying is an efficient drying technique for the retention of citrus by-product polyphenols when it is compared with hot-air drying being conducted at relatively lower temperatures (<60°C). [39] As the hot-air drying temperature decreases, the required drying time increases, resulting in the degradation of some phenolic compounds due to enzymatic oxidation. On the other hand, hot-air drying at high temperatures (>70°C) is more efficient than the freeze drying technique. [10,39] High temperatures may result in the denaturation of the enzymes being implicated in the oxidation of phenolic compounds and promote the liberation of the bound polyphenols [9,45] (Fig. 2). Therefore, greater TPC yields are obtained compared to freeze drying.

Hot-air drying

During hot-air drying, heat is transferred to the material by convection and dehydration takes place. In general, hot-air drying requires shorter drying times and energy consumption than freeze drying. [10] The effect of hot-air drying at different operating conditions on the recovery of total and individual polyphenols has been extensively studied and compared with other drying techniques. [10,37,39,43,44,46] Previous studies have shown that hot-air drying could be effectively applied to citrus by-products for high recovery of polyphenols and antioxidants. [9,10,46] However, three parameters should be considered when hot-air drying is applied, namely, (i) drying temperature, (ii) drying time, and (iii) air velocity, since undesirable hot-air drying conditions may lead to degradation and significant loss of polyphenols. During hot-air drying polyphenol enzymatic and nonenzymatic degradation may take place. [20] Hot-air drying results in the disruption of cell walls and the liberation of bound polyphenols. At the same time, oxidative and hydrolytic enzymes are released that can degrade the liberated phenolic compounds. As the applied temperature increases, the released enzymes are inactivated. At high temperatures, a nonenzymatic polyphenol degradation/conversion takes place and a discoloration of the dried material occurs [10,39,45–48], which has been attributed to the formation of Maillard reaction products. [49]

Lou et al. [45] indicated that the TPC, phenolic acid, and flavonoid content of immature calamondin peels dried by hot air at 150°C was significantly higher than those dried at lower temperatures or freeze dried. However, at these conditions, the recovery of some individual compounds (3′,5′-Di-C-β-glucopyranosylphloretin, and hesperidin) was significantly lower (90 and 290 mg/100 g d.e., respectively) compared to the peels dried by freeze drying (4322 and 430 mg/100 g d.e., respectively). During hot-air drying at high temperatures, some phenolic compounds might be degraded and converted into other products since high temperatures result in the polyphenol deglycosylation. [50] Interestingly, the authors found varying effects on the retention of individual phenolic acids. For instance, ferulic acid, *p*-coumaric acid, and gallic acid were not detected in the extracts of the freeze-dried peels but were found in the extracts of the peels dried by hot air, while the opposite was found for the caffeic acid. [45] The higher ferulic acid, *p*-coumaric acid, and gallic acid yields obtained from the hot-air dried peels could be attributed to the thermal destruction of cell walls, to the liberation of the bound phenolic acids, as well as to the conversion of some phenolic acids to others due to the heat treatment. [50] The reduced caffeic acid yields could be attributed to the oxidation and transformation of caffeic to ferulic acid. Ferulic acid is biosynthesized from caffeic acid by O-methylation, a reaction that increases as the temperature increases. [51,52] Similarly, Papoutsis et al. [10] found that hot-air drying conditions had varying effects on the recovery of phenolic acids from lemon by-products. Higher gallic acid content was obtained from the lemon by-products dried at 110°C (8.71 μg/mL) than those dried at 70 and 90°C (1.10 and 4.69 μg/mL, respectively), while *p*-coumaric acid content was higher in the by-products dried at 90 and 70°C (1.03 and 0.99 μg/mL, respectively), than those dried at 110°C (0.71 μg/mL).

Ghanem Romdhane et al. [37] investigated the effect of different hot-air temperatures (40–60°C) on the degradation kinetics of TPC and flavonoids of lemon peels. In the range of 40–60°C, a significant reduction in the TPC and flavonoid contents were observed, with the temperature of 40°C resulting in the highest reduction of TPC and flavonoid contents (72% and 20%, respectively). The degradation kinetics of both parameters were fitted to a first-order equation. These findings revealed that phenolic compounds in citrus by-products are susceptible to degradation when the materials are dried at low temperatures for a long time; thus, drying temperature exhibits an important influence on the degradation rates of polyphenols. During citrus by-product drying at low temperatures, a rapid polyphenol degradation may occur due to the high PPO activity during the first hour of the process. [53] Ghanem Romdhane et al. [54] investigated the kinetics of lemon peel polyphenol degradation during osmo-dehydration (50–70%w/w) at different temperatures (30–50°C) followed by hot-air drying (at 40 and 60°C). A significant decrease (70–80%) in the TPC of the osmo-dehydrated lemon peels was observed with increasing sucrose concentration (from 50 to 70%w/w) and sucrose solution temperature (from 30 to 50°C). However, following osmo-dehydration, drying citrus by-products at 40 or 60°C had no significant effect on the TPC. The polyphenol loss that took place during osmo-dehydration was attributed to the osmotic driving force, which promotes the migration of soluble phenolic compounds from the peels to the osmotic solution. [54] Future studies should be conducted in order to investigate the exact mechanism involved in the retention of polyphenols during drying after osmo-dehydration.

Hot-air drying, being a lower energy consumption method than freeze drying, could be effectively applied for drying citrus by-products. However, the drying conditions should be carefully selected for avoiding polyphenol degradation, since different drying temperatures show varying effects on the different classes of citrus by-product polyphenols. In conclusion, hot-air drying at high temperatures may promote the recovery of phenolic compounds from citrus by-products, while hot-air drying at low temperatures might have adverse effects.

Sun drying

Sun drying is a low-cost drying technique since it does not require any expensive instrument installation; however, it is dependent on various environmental parameters, including relative humidity, temperature, air velocity, and solar intensity among others. During sun drying, microbial contamination of the citrus by-products may occur contributing to the loss of the dried material quality. Moreover, sun drying requires prolonged drying periods, which may negatively affect the retention of polyphenols in the dried material. [9,34] Sun et al. [9] mentioned that the flavonoid and phenolic acid contents of different immature citrus species dried by sun drying (for 3 days) were significantly lower than those dried by freeze drying (for 12 hours) or hot-air drying at 60°C (for 10 hours). The reduced polyphenol retention in the sun-dried citrus by-products could be attributed to the polyphenol enzymatic oxidation (PPO) due to the presence of oxygen, as well as to the photo-oxidation of some phenolic compounds. When citrus by-products are exposed to the sun, the UV irradiation may induce the formation of free radicals, which may be scavenged by the phenolic compounds, resulting in polyphenol oxidation. [21] Even though sun drying is a low-cost drying technique, it requires extended drying times that might negatively affect the concentration of citrus by-product polyphenols.

Vacuum drying

Vacuum drying has been applied for drying various fruits and vegetables. [55,56] However, it has been rarely used for the dehydration of citrus by-products. During vacuum drying, the moisture removal from the raw material takes place under low pressure in the absence of oxygen, preventing the enzymatic oxidation of phenolics [56] (Fig. 2). A recent study compared the effect of vacuum drying operating at different conditions with hot-air drying and freeze drying on the retention of polyphenols of lemon by-products. [10] Vacuum drying temperature significantly affected the recovery of different classes of lemon by-product polyphenols. [10] During vacuum drying, the heat energy may cleave the polyphenols being linked to the cell wall polymers, facilitating higher recovery of the free forms; however, temperatures higher than the optimum may result in the degradation of the heat sensitive phenolic compounds. [16,23] For instance, high rutin and *p*-coumaric acid extraction yields were achieved by drying lemon by-products with vacuum drying at 70°C (137.04 and 1.69 μg/mL, respectively), which were significantly higher than those obtained by freeze drying (119.29 and 1.10 μg/mL, respectively) or hot-air drying at 70°C (107.31 and 0.99 μg/mL, respectively). [10] These results indicate that during the thermal processing of citrus by-products, some bound phenolic compounds might be liberated. However, drying temperatures higher than the optimum might lead to polyphenol degradation due to cleavage of covalent bonds. [57]

According to the previous results, comparing vacuum drying with hot-air drying, it could be concluded that during hot-air drying some polyphenols might be oxidized due to material exposure to oxygen. The advantages of vacuum drying over other drying techniques include: (i) less energy consumption compared to freeze drying, (ii) lower processing temperatures compared to hot-air drying, therefore less polyphenol degradation due to heat, and (iii) prevention of polyphenol enzymatic degradation, since it takes place in oxygen absence [55] (Fig. 2). However, during vacuum drying at high temperatures, a discoloration of the dried material may occur. [10]

Microwave drying

Microwaves have been used in several food procedures including pasteurization and cooking, among others since they result in substantially reduced processing times. [58] During microwave drying, the microwave power is absorbed by the peels and leads to internal water heating and moisture evaporation. During this process, the solubility of the pectic material of the middle lamellae increases, facilitating cell wall rupture. [59] The microwave processing time decreases as the microwave power increases and depends on the citrus species. [59] Citrus species with thicker peels require shorter microwave drying times. [59] Recently, the effect of microwave drying on the retention of citrus by-product polyphenols was investigated. [42,59,60] Microwave drying may have varying effects on the retention of TPC of the different citrus species. For instance, microwave drying negatively affected the retention of TPC in mandarin and lemon peels, while it enhanced the TPC recovery from orange peels. [59] Bejar et al. [60] investigated the effect of different microwave drying conditions (power ranging between 100 and 850 W) on the retention of TPC of orange peels. The retention of TPC increased as the microwave power increased to an optimum level and then declined. The power of 450 W was found to be optimum for the recovery of TPC (1.88 g caffeic acid equivalents/100 g dry weight). Higher microwave powers negatively affected the recovery of TPC. This effect could be due to the thermal degradation of some phenols because of the heat developed during the microwave process. Microwave powers lower than 450 W were reported as unfavorable for the recovery of TPC probably due to the oxidation of some of the phenolic compounds from oxygen exposure for a long time. [60]

Indeed, microwave drying could be employed for drying citrus by-products [58]; however, studies investigating and determining the effect of different microwave operating conditions on the retention of individual phenolic compounds in different citrus species should be conducted.

High-speed drying

Senevirathne et al. [19] used high-speed drying, which is a rapid and economical technique, for drying citrus by-products. Fig. 3 illustrates the diagram of a high-speed drier. During high-speed drying, the plant material is placed into the sample compartment of the system. The loaded material is conducted upwards to the heat transmitting wall by the rotating cyclone fin and it is held against the heating wall in a thin film by centrifugal force. The wall is heated by the steam supplied by the steam generator. The waste steam from the process, which has high moisture content, is conducted to the super-heat steam generator, where this waste steam is burnt at a temperature higher than 700°C and emitted under high temperature oxidization in an odorless condition. When the moisture content of the dried material reaches a specific value, a thermo-sensor sends a signal that automatically stops the unit, and the dried product can be obtained. [19]

High-speed drying required significantly lower drying time (90 minutes) compared to freeze drying (24 hours) and the extracts obtained from the high-speed dried citrus by-products showed a high content of polymethoxylated flavones and flavanones, as well as strong radical scavenging activity and lipid peroxidation inhibition. [19] High-speed drying requires slightly longer drying times compared to microwave drying. However, during microwave drying, a thermal degradation of some polyphenols may occur, because of the heat developed during the microwave process. [60] Moreover, high microwave powers may lead to the formation of Maillard reaction products, resulting in a color change of the dried material. [34,49]

Infrared and far-infrared drying

Infrared is an electromagnetic irradiation and based on its wavelength is divided in near-infrared (NIR; 0.78–1.4 mm), middle-infrared (MIR; 1.4–3 mm), and far-infrared (FIR; 3–1000 mm). [61] Infrared drying has been employed for the dehydration of food products and is popular because of its various advantages, including energy savings, shorter drying time than freeze drying, high-quality dried products, uniform temperature distribution, and clean operational environment. [61,62]

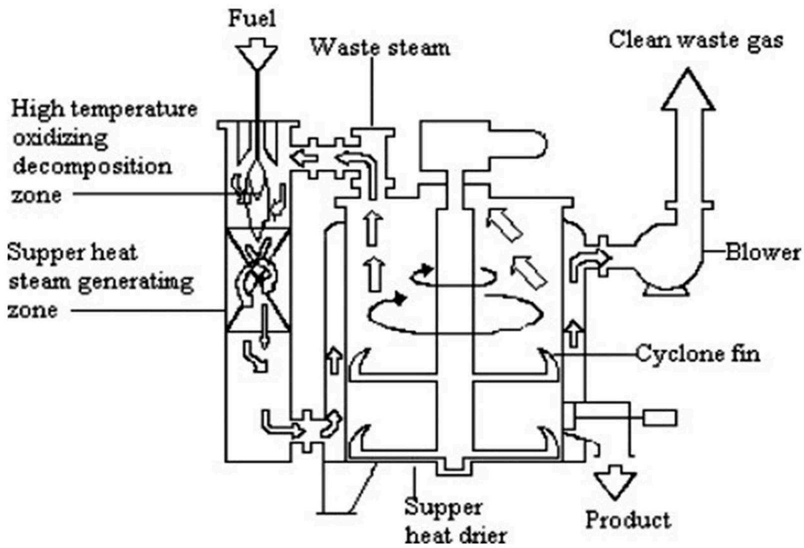

Figure 3. Flow diagram of a high-speed drier (Model: Okadora Korea). Reprinted from J. Food Eng., 92, Senevirathne, M.; Jeon, Y.J.; Ha, J.H.; Kim, S.H., Effective drying of citrus by-product by high-speed drying: A novel drying technique and their antioxidant activity, 2009, 157-163, Copyright (2017), with permission from Elsevier.

Bejar et al. [17] investigated the effect of different infrared drying temperatures on the recovery of TPC from orange peels and found that the TPC yields of the extracts obtained from the infrared dried peels were significantly lower than those obtained from the fresh ones. It was mentioned that in the dried materials, all the plant cell components adhered together decreasing the surface area being exposed to the solvent. [17] Therefore, the accessibility of the analyte to the extraction solvent decreased resulting in reduced TPC yields. [63] Senevirathne et al. [18] employed far-infrared drying at different temperatures (40, 50, 60, 70, and 80°C) for converting wet citrus press-cakes into dried and obtained a varied effect on the individual and total phenolic compounds. For instance, the TPC, nobiletin, sinensetin, 3′,4′,7,8-tetramethoxy flavone, and tangeretin contents of the extracts obtained from the far-infrared dried cakes were lower than those obtained from the freeze-dried ones. However, higher retention of neohesperidin, narirutin, and quercetagetin was observed in the samples dried by far-infrared drying compared to those dried by freeze drying. [18] The heat generated due to the far-infrared irradiation may lead to the cleavage of some polyphenols being linked to the cell wall polymers, facilitating higher recovery of the free forms; however, at the same time the liberated polyphenols may be degraded due to the formation of reactive oxygen species as a result of far-infrared irradiation. [18]

Post-drying treatments for liberating bound polyphenols

Recent studies have indicated that higher extraction yields of individual phenolic compounds and antioxidant capacity could be achieved by further applying heat (conventional oven and microwave oven) or irradiation (UV and electron-beam) treatments on the dried citrus by-products prior to extraction [22,23,25,26,64] (Table 5).

Table 5. Post-drying treatments applied on dried citrus for liberating phenolic compounds and enhancing the polyphenol content of the extracts.

After drying treatments			
Citrus species	Technique	Comments	Refs
Lemon by-products	• Freeze drying followed by UV-C treatment.	• UV-C irradiation of 19 kJ/m^2 resulted in extracts with higher TPC than the control, while UV-C irradiation of 180 kJ/m^2 resulted in extracts with higher total flavonoid and proanthocyanidin contents than the control.	[25]
Lemon by-products	• Freeze drying followed by microwave treatment.	• Appropriate microwave treatment resulted in the increase of the TPC, total flavonoids, proanthocyanidins, and total antioxidant capacity of the extracts.	[24]
Mandarin by-products	• Sun drying followed by microwave treatment.	• Appropriate microwave treatment could liberate and activate the bound phenolic compounds of the mandarin by-products.	[16,23]
Citrus unshiu by-products	• Freeze drying followed by electron-beam irradiation treatment.	• The TPC and radical scavenging activity of the freeze-dried by-product powder treated at 37.9 kGy, significantly increased compared to the non-irradiated control.	[22]
Huyou peels	• Hot-air dried peels followed by heat treatment (electric oven).	• Heat treatment resulted in the increase of the free phenolic acid content, and the decrease of ester, glycoside, and ester-bound phenolic acids. • Heat treatment resulted in the decreased of the flavanone glycosides. • After heat treatment the antioxidant capacity of the extracts increased.	[64]
Citrus unshiu peels	• Air-drying followed by heat treatment (electric muffle furnace).	• The extracts of the dried citrus peels treated at 150°C had higher TPC and radical scavenging activity compared to those obtained from the non-heated control. • Several low molecular weight phenolic compounds such as ferulic acid, 5-hydroxyvaleric acid, 2,3-diacetyl-1-phenylnaphthalene, and vanillic acid etc. were newly formed in the peels treated at 150°C.	[26]

Heat treatment

Phenolic compounds contained in citrus peels include phenolic acids (hydroxyl cinnamic acids) [65] and flavonoids (flavanones, flavones, and flavonols). [66] In citrus peels, phenolic acids are often linked to various plant components through ester and glucoside bonds [64], whereas flavonoids can be present in the free (aglycones) or bound (glycoside) forms, with the free forms having higher antioxidant properties compared to the bound ones. The application of heat on the dried citrus by-products may enhance the liberation of polyphenols by breaking down both ester and glucoside bonds. [23,24,26] Both ester and glucoside bonds are types of covalent bonds and they tend to be very stable because the energies required to break them are higher than the thermal energy available at ambient temperature. [67] Therefore, as the temperature increases, the thermal energy increases facilitating the cleavage of covalent bonds, hence the liberation of bound polyphenols. However, high temperatures may result in the formation of Maillard reaction products [49], changing the color of the material. [34] To date, heat has been applied either by the use of conventional or microwave ovens. The advantages and disadvantages of each method are discussed in the following sections.

Conventional oven

Jeong et al. [26] treated the dried peels of *Citrus unshiu* at different temperatures (50, 100, or 150°C) for different intervals using an electric muffle furnace and found that both temperature and time had a significant effect on their TPC. The extracts of the dried citrus peels treated at 150°C for 40 minutes had higher TPC and radical scavenging activity (171.0 µM and 59.73%, respectively) compared to the unheated control (71.8 µM and 29.64%). The GC-MS chromatograms revealed the formation of several low molecular weight phenolic compounds in the heat treated samples, including 2,3-diacetyl-1-phenylnaphthalene, ferulic acid, vanillic acid among others (Fig. 4). During heat treatment, both ester and glucoside bonds can be broken down resulting

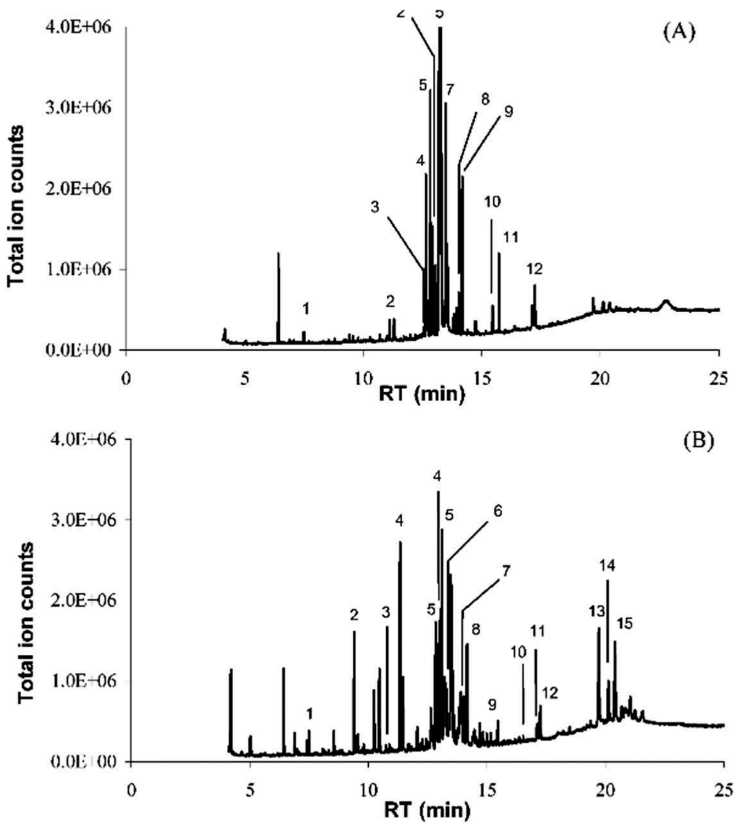

Figure 4. Typical gas chromatography of 70% ethanol extracts from citrus peels nonheated (A) and heated (B) at 150 °C for 30 min. Peaks in (A): 1, bishydroxybutanedioic acid; 2, 2-azathianthrene; 3, 2-oxybenzoic acid; 4, 1H-indole-3-carboxaldehyde; 5, arabinofuranose; 6, α-DL-arabinofura-noside; 7, β-L-arabinopyranose; 8, glucopyranose; 9, palmitic acid; 10, 2,4-bishydroxybenzaldehyde; 11, stearic acid. Peaks in (B): 1, bishy-droxybutanedioic acid; 2, 2,3-diacetyl-1-phenylnaphthalene; 3, 1,2-ben-zenedicar-boxylic acid ethyl ester; 4, 2-azathianthrene; 5, arabinofuranose; 6, β-D-galactofuranose; 7, β-L-arabi-nopyranose; 8, glucopyranose; 9, palmitic acid; 10, ferulic acid; 11, p-hydroxybenzaldoxime; 12, stearic acid; 13, D-ribofuranose; 14, glucofuranoside; 15, β-D-galactofuranoside. Reprinted from J. Agric. Food Chem. with permission from Jeong, S.M.; Kim, S.Y.; Kim, D.R.; Jo, S.C.; Nam, K.C.; Ahn, D.U.; Lee, S.C. Effect of heat treatment on the antioxidant activity of extracts from citrus peels. 2004, 52, 3389-3393. Copyright (2017) American Chemical Society.

in the liberation of the bound polyphenols. At the same time, heat may lead to polyphenol transformation explaining why the content of some phenolic compounds increases while the content of others decreases. [27] Similar results were reported by Xu et al. [64] who treated the hot-air dried peels of huyou (*Citrus paradisi* Changshanhuyou) at different temperatures and intervals using a conventional oven. The authors reported that heat treatment resulted in the release of phenolic acids, since the free fraction of both benzoic and cinnamic phenolic acids increased, while ester, glycoside, and ester-bound fractions decreased, indicating that heat treatment of dried citrus by-products facilitates the breakdown of both ester and glucoside bonds. On the other hand, heat treatment negatively affected the content of flavanone glycosides (narirutin, naringin, hesperidin, and neohesperidin), implying that flavanones are vulnerable to heat treatment. According to the previous results, heat treatment could be effectively applied for liberating and enhancing the recovery yields of polyphenols from citrus by-products. However, high temperatures may lead to the degradation of some flavonoid compounds. Therefore, the heat treatment conditions should be carefully selected considering the type of polyphenols are aimed to be extracted.

Microwave

Microwave ovens have been mainly used for the extraction of bioactive compounds from plant matrices. [68,69] Recent studies revealed that microwave energy could be effectively applied for enhancing the recovery of polyphenols and antioxidants from citrus by-products. [16,23,24] Due to their electromagnetic nature, microwave irradiation (frequency of 0.3–300 GHz) results in a reduced heating time compared to conventional heating techniques, because of the heating of individual elements of a material. [23,70] During the microwave treatment, the heat being generated in the dried material results in the cleavage of covalent bonds, which lead to the liberation of the bound polyphenols. [60]

Hayat et al. [23] treated the sun-dried citrus by-products with different microwave powers for different intervals. Both microwave power and treatment time significantly affected the contents of individual phenolic acids. The free forms of phenolic acids increased with increasing microwave power, whereas the bound forms of phenolic acids declined, revealing liberation of the bound forms of phenolic acids due to the heat caused by the microwave energy. The authors made the assumption that the release of phenolic acids could be attributed either to the selective heating of some of the individual phenolic acids in the microwave field or to the physical forces acting between phenolic acids and plant matrix. Phenolic acids are linked to plant matrix through covalent bonds. The application of microwave irradiation results in the heat generation that leads to the cleavage of the bound phenolics. [67] Future studies investigating the exact mechanism implicated in the liberation of individual phenolic compounds from the plant tissues should be conducted for elucidating the mechanism of action.

Apart from liberating phenolic acids, microwave treatment could be effectively applied for enhancing the recovery of flavonoids (flavanols, flavanones, and flavonols) from dried citrus by-products. [23] Microwave treatment of 250 W for 10 minutes was proved to significantly enhance the recovery of catechin, naringin, rutin, kaempferol, and hesperidin from the sun-dried citrus by-products. [23] However, the recovery yields of the individual flavonoids decreased when the citrus by-products were treated for a longer time at 250 W. The heat generated as a result of the microwave irradiation results in the cleavage of the bound polyphenols and the cell wall rupture, leading to greater polyphenol yields. [16,23] At the same time, prolonged exposure to microwave irradiation may result in the thermal degradation or conversion of the liberated flavonoids. [71] These results are in accordance with a recent study which reported that appropriate microwave treatment of the freeze-dried lemon by-products resulted in higher recovery of TPC, total flavonoid content (TF), proanthocyanidins, and antioxidants. [24] The increased antioxidant capacity of the extracts obtained from the microwave-treated citrus by-products could be due to the release of the bound polyphenols, as well as to the formation of Maillard reaction products, which are compounds with antioxidant properties. [71]

Irradiation

Ultraviolet C (UV-C) irradiation

UV-C (200–280 nm) is a nonionizing, electromagnetic irradiation that has been shown to promote the accumulation of polyphenols and antioxidants in fresh fruits and vegetables. [72,73] The effect of UV-C irradiation on the recovery of polyphenols and antioxidants from freeze-dried lemon by-product was examined. [25] Freeze-dried lemon by-products were subjected to different UV-C irradiations, and the results showed that the recovery of TPC and TF increased by 19% and 28%, respectively, when the dried by-products were treated with UV-C irradiation of 19 and 180 kJ/m^2, respectively, compared to the control (untreated dried by-products). The higher TPC and TF yields could be attributed to the cleavage of the bound polyphenols caused by heating effects due to the transfer of energy of many photons. Given that the previous study reported the effect of UV-C irradiation only on the recovery of total polyphenols, more studies should be conducted for elucidating the mechanism and UV-C effect on the recovery of individual phenolic compounds from dried citrus by-products. Although UV-C irradiation may positively affect the recovery of polyphenols from citrus by-products, a number of adverse effects of UV irradiation on human health have been identified and should be considered when this technology is employed. Personnel exposed to UV irradiation may suffer eye damage, erythema, and immunosuppression, while chronic exposure of the skin to the UV irradiation may cause skin cancer. [74,75]

Electron-beam irradiation (EBI)

EBI is a low dose ionizing irradiation that has been used in the food and pharmaceutical industries for eliminating microbial contamination. [76] EBI has been characterized as a safe technology with no potential health risks. [76] The effect of EBI on the phytochemicals of fresh plant tissues has been previously reported. [77] Kim et al. [22] treated the freeze-dried by-product of *C. unshiu* with five EBI dosages (3.0, 5.6, 10.9, 25.5, and 37.9 kGy) and showed that when the freeze-dried powder was treated at 37.9 kGy, the TPC, and the radical scavenging activity of the water extracts increased from 6543.2 to 7405.4 mM, and 37.6% to 52.9%, respectively, compared to the control (non-irradiated dried by-product). These results were attributed to the liberation of some phenolic compounds due to heating effect in the treated dried by-products because of the EBI since previous studies have reported the liberation of individual phenolic compounds (phenolic acids) as a result of heat treatment. [23] However, during electron-beam irradiation, the bound polyphenols might be liberated due to the energy of individual photons, which is enough to break chemical bonds. [28] Future studies should be conducted in order to elucidate the mechanism implicated in the liberation and recovery of individual polyphenols (phenolic acids and flavonoids) from plant materials during EBI treatment.

Conclusions and future trends

Citrus by-products are usually exposed to undesirable environmental conditions for a long time after juice production and an important reduction in their polyphenol content may occur during this period. Future studies should investigate and determine the level of polyphenol degradation as a result of citrus by-product exposure to undesirable environmental conditions prior to material dehydration. Drying is a crucial but not compulsory step that precedes the extraction process of bioactive compounds from citrus by-products. The advantages of citrus by-product dehydration include (i) reduced packaging volume, hence lower transportation cost and storage space requirements, (ii) prevention of material spoilage, and (iii) prevention of polyphenol enzymatic degradation.

Several parameters should be considered for the selection of the drying technique, including energy consumption, residual moisture content in the dried material, and retention of bioactive compounds in the dried product. Several methods have been employed for drying citrus by-products, such as freeze drying, hot-air drying, vacuum drying, sun drying, with each drying technique having its own advantages and limitations. Generally, freeze drying is considered as the most efficient but expensive drying technique. Freeze drying results in high retention levels of flavonoids and phenolic acids in citrus by-products. However, a number of studies have indicated that hot-air drying at high temperatures may result in dried citrus materials with higher phenolic acid and flavonoid contents than those dried by freeze drying. For instance, ferulic acid, *p*-coumaric acid, and gallic acid recovery could be higher in the extracts of citrus by-products dried by hot-air drying than those dried by freeze drying. Although hot-air drying at high temperatures may promote the recovery of some phenolic compounds, lower hot-air drying temperatures may result in a significant enzymatic degradation of polyphenols, due to material exposure to oxygen for long times. When citrus wastes are dried at low temperatures, a rapid polyphenol degradation may occur during the first hour of the process. This loss might be prevented by the application of some treatments prior to hot-air drying, such as osmotic dehydration of the citrus by-product. Future studies should take into consideration the periods during hot-air drying when polyphenols are degraded and try to minimize polyphenol reduction by applying treatments prior to extraction for inactivating enzymes implicated in polyphenol degradation. Vacuum drying is a technique that takes place in the absence of oxygen and at lower temperatures compared to hot-air drying, preventing the degradation of polyphenols being vulnerable to oxygen and heat exposure. Although several comparison studies have been conducted on citrus by-products, only a few have included the effect of vacuum drying on polyphenol retention.

To date, some new drying techniques have been applied for drying citrus by-products, including, microwave drying, infrared drying, far-infrared drying, and high-speed drying. Among others, high-speed drying seems to be the most effective since it results in a dried product with low moisture content, and high retention of polyphenols in a significantly shorter time than freeze drying. On the other hand, although both microwave and infrared drying require shorter times for drying citrus by-products compared to freeze drying, the retention of polyphenols in the microwave and infrared dried citrus by-products is significantly lower compared to those dried by freeze drying. Some comparisons of different drying methods have been performed; however, they remain partial since only two or three methods are compared in the same work. Although several studies have been conducted investigating the effect of different drying treatments on the retention of polyphenols in citrus by-products, only a few have provided information about the degradation kinetics of individual phenolic compounds during the drying process. The degradation kinetics of individual phenolic compounds during drying process will facilitate the development of the optimum drying conditions for high retention of polyphenols in the dried material.

Recently, studies have been conducted investigating the effect of different treatments of the dried citrus by-products on the liberation of bound polyphenols. Microwave could be effectively used for liberating phenolic acids from dried citrus by-products; however, the exact mechanism implicated in the liberation of individual phenolic compounds from the plant tissues needs clarification. Other treatments, such as UV-C or EBI could be applied on dried citrus by-products for enhancing their phenolic content and antioxidant activities. Studies investigating the effect of these treatments on the retention of individual polyphenols are recommended.

Disclosure statement

The authors declare no conflict of interest.

Funding

The author thanks to the University of Newcastle and Australian Research Council (ARC) Training Centre for Food and Beverage Supply Chain Optimisation (IC140100032) for research funding the PhD scholarship to Konstantinos Papoutsis.

ORCID

Konstantinos Papoutsis http://orcid.org/0000-0003-0126-1619

References

[1] FAO, Intergovernmental Group on Citrus Fruits. A Subsidiary Body of the FAO Committee on Commodity Problems (CCP) In, Rome, **2016**. http://www.fao.org/3/a-i5558e.pdf 2016 (accessed Jan 03, 2017).

[2] Lanuzza, F.; Mondello, F.; Tripodo, M. M. Studies about the Utilization of Citrus Wastes in View of Environment Protection. In *Pathways to Environmental Sustainability: Methodologies and Experiences*; Salomone, R., Saija, G., Eds.; Springer International Publishing: Switzerland, **2014**; pp 147–156.

[3] Islam, M. Z.; Kitamura, Y.; Kokawa, M.; Monalisa, K.; Tsai, F. H.; Miyamura, S. Effects of Micro Wet Milling and Vacuum Spray Drying on the Physicochemical and Antioxidant Properties of Orange (*Citrus unshiu*) Juice with Pulp Powder. *Food Bioprod. Process*. **2017**, 101, 132–144.

[4] Dhanavade, M. J.; Jalkute, C. B.; Ghosh, J. S.; Sonawane, K. D. Study Antimicrobial Activity of Lemon (*Citrus lemon* L.) Peel Extract. *Br. J. Pharmacol. Toxicol.* **2011**, 2, 119–122.

[5] Ortuño, A.; Báidez, A.; Gómez, P.; Arcas, M. C.; Porras, I.; García-Lidón, A.; Río, J. A. D. *Citrus paradisi* and *Citrus sinensis* Flavonoids: Their Influence in the Defence Mechanism against *Penicillium Digitatum*. *Food Chem*. **2006**, 98, 351–358.
[6] Rawson, N. E.; Ho, C. T.; Li, S. Efficacious Anti-Cancer Property of Flavonoids from Citrus Peels. *Food Sci. Hum. Well.* **2014**, 3, 104–109.
[7] Papoutsis, K.; Pristijono, P.; Golding, J. B.; Stathopoulos, C. E.; Bowyer, M. C.; Scarlett, C. J.; Vuong, Q. V. Optimisation of Aqueous Extraction Conditions for the Recovery of Phenolic Compounds and Antioxidants from Lemon Pomace. *Int. J. Food Sci. Tech*. **2016**, 51, 2009–2018.
[8] Wilmsen, P. K.; Spada, D. S.; Salvador, M. Antioxidant Activity of the Flavonoid Hesperidin in Chemical and Biological Systems. *J. Agric. Food Chem*. **2005**, 53, 4757–4761.
[9] Sun, Y.; Shen, Y.; Liu, D.; Ye, X. Effects of Drying Methods on Phytochemical Compounds and Antioxidant Activity of Physiologically Dropped Un-Matured Citrus Fruits. *LWT - Food Sci. Tech*. **2015**, 60, 1269–1275.
[10] Papoutsis, K.; Pristijono, P.; Golding, J. B.; Stathopoulos, C. E.; Bowyer, M. C.; Scarlett, C. J.; Vuong, Q. V. Effect of Vacuum-Drying, Hot Air-Drying and Freeze-Drying on Polyphenols and Antioxidant Capacity of Lemon (*Citrus limon*) Pomace Aqueous Extracts. *Int. J. Food Sci. Tech*. **2017**, 52, 880–887.
[11] Jenkins, G. I. Signal Transduction in Responses to UV-B Radiation. *Annu. Rev. Plant Biol.* **2009**, 60, 407–431.
[12] Dai, J.; Mumper, R. J. Plant Phenolics: Extraction, Analysis and Their Antioxidant and Anticancer Properties. *Molecules*. **2010**, 15, 7313–7352.
[13] González-Molina, E.; Domínguez-Perles, R.; Moreno, D. A.; García-Viguera, C. Natural Bioactive Compounds of *Citrus limon* for Food and Health. *J. Pharm. Biomed. Anal*. **2010**, 51, 327–345.
[14] Benavente-García, O.; Castillo, J.; Marin, F. R.; Ortuño, A.; Del Río, J. A. Uses and Properties of Citrus Flavonoids. *J. Agric. Food Chem*. **1997**, 45, 4505–4515.
[15] Robbins, R. J. Phenolic Acids in Foods: An Overview of Analytical Methodology. *J. Agric. Food Chem*. **2003**, 51, 2866–2887.
[16] Hayat, K.; Zhang, X.; Farooq, U.; Abbas, S.; Xia, S.; Jia, C.; Zhong, F.; Zhang, J. Effect of Microwave Treatment on Phenolic Content and Antioxidant Activity of Citrus Mandarin Pomace. *Food Chem*. **2010**, 123, 423–429.
[17] Bejar, K. A.; Ghanem, N.; Mihoubi, D.; Kechaou, N.; Boudhrioua Mihoubi, N. Effect of Infrared Drying on Drying Kinetics, Color, Total Phenols and Water and Oil Holding Capacities of Orange (*Citrus sinensis*) Peel and Leaves. *Int. J. Food Eng*. **2011**, 7, DOI: 10.2202/1556-3758.2222.
[18] Senevirathne, M.; Kim, S. H.; Kim, Y. D.; Oh, C. K.; Oh, M. C.; Ahn, C. B.; Je, J. Y.; Lee, W. W.; Jeon, Y. J. Effect of Far-Infrared Radiation Drying of Citrus Press-Cakes on Free Radical Scavenging and Antioxidant Activities. *J. Food Eng*. **2010**, 97, 168–176.
[19] Senevirathne, M.; Jeon, Y. J.; Ha, J. H.; Kim, S. H. Effective Drying of Citrus By-Product by High Speed Drying: A Novel Drying Technique and Their Antioxidant Activity. *J. Food Eng.* **2009**, 92, 157–163.
[20] Abhay, S. M.; Hii, C. L.; Law, C. L.; Suzannah, S.; Djaeni, M. Effect of Hot-Air Drying Temperature on the Polyphenol Content and the Sensory Properties of Cocoa Beans. *Int. Food Res. J*. **2016**, 23, 1479–1484.
[21] Volf, I.; Ignat, I.; Neamtu, M.; Popa, V. I. Thermal Stability, Antioxidant Activity, and Photo-Oxidation of Natural Polyphenols. *Chem. Pap*. **2014**, 68, 121–129.
[22] Kim, J. W.; Lee, B. C.; Lee, J. H.; Nam, K. C.; Lee, S. C. Effect of Electron-Beam Irradiation on the Antioxidant Activity of Extracts from *Citrus unshiu* Pomaces. *Radiat. Phys. Chem*. **2008**, 77, 87–91.
[23] Hayat, K.; Zhang, X.; Chen, H.; Xia, S.; Jia, C.; Zhong, F. Liberation and Separation of Phenolic Compounds from Citrus Mandarin Peels by Microwave Heating and Its Effect on Antioxidant Activity. *Sep. Purif. Technol*. **2010**, 73, 371–376.

[24] Papoutsis, K.; Pristijono, P.; Golding, J. B.; Stathopoulos, C. E.; Bowyer, M. C.; Scarlett, C. J.; Vuong, Q. V. Enhancement of the Total Phenolic Compounds and Antioxidant Activity of Aqueous *Citrus limon* L. Pomace Extract Using Microwave Pretreatment on the Dry Powder. *J. Food Process. Preserv*. **2016**. DOI: 10.1111/jfpp.13152.
[25] Papoutsis, K.; Vuong, Q. V.; Pristijono, P.; Golding, J. B.; Bowyer, M. C.; Scarlett, C. J.; Stathopoulos, C. E. Enhancing the Total Phenolic Content and Antioxidants of Lemon Pomace Aqueous Extracts by Applying UV-C Irradiation to the Dried Powder. *Foods*. **2016**, 5, DOI: 10.3390/foods5030055.
[26] Jeong, S. M.; Kim, S. Y.; Kim, D. R.; Jo, S. C.; Nam, K. C.; Ahn, D. U.; Lee, S. C. Effect of Heat Treatment on the Antioxidant Activity of Extracts from Citrus Peels. *J. Agric. Food Chem*. **2004**, 52, 3389–3393.
[27] Buchner, N.; Krumbein, A.; Rohn, S.; Kroh, L. W. Effect of Thermal Processing on the Flavonols Rutin and Quercetin. *Rapid Commun. Mass Spectrom*. **2006**, 20, 3229–3235.
[28] Tokunaga, T.; Narushima, T.; Yonezawa, T.; Sudo, T.; Okubo, S.; Komatsubara, S.; Sasaki, K.; Yamamoto, T. Temperature Distributions of Electron Beam-Irradiated Samples by Scanning Electron Microscopy. *J. Microsc*. **2012**, 248, 228–233.
[29] M'Hiri, N.; Ioannou, I.; Ghoul, M.; Boudhrioua, N. M. Extraction Methods of Citrus Peel Phenolic Compounds. *Food Rev. Int*. **2014**, 30, 265–290.
[30] Sharma, K.; Mahato, N.; Cho, M. H.; Lee, Y. R. Converting Citrus Wastes into Value-Added Products: Economic and Environmently Friendly Approaches. *Nutrition*. **2017**, 34, 29–46.
[31] Putnik, P.; Bursać Kovačević, D.; Režek Jambrak, A.; Barba, F.; Cravotto, G.; Binello, A.; Lorenzo, J.; Shpigelman, A. Innovative "Green" and Novel Strategies for the Extraction of Bioactive Added Value Compounds from Citrus Wastes – A Review. *Molecules*. **2017**, 22, DOI: 10.3390/molecules22050680.
[32] Sui, Y.; Yang, J.; Ye, Q.; Li, H.; Wang, H. Infrared, Convective, and Sequential Infrared and Convective Drying of Wine Grape Pomace. *Dry. Technol*. **2014**, 32, 686–694.
[33] Delele, M. A.; Weigler, F.; Mellmann, J. Advances in the Application of a Rotary Dryer for Drying of Agricultural Products: A Review. *Dry. Technol*. **2015**, 33, 541–558.
[34] Chen, H. H.; Hernandez, C. E.; Huang, T. C. A Study of the Drying Effect on Lemon Slices Using a Closed-Type Solar Dryer. *Sol. Energy*. **2005**, 78, 97–103.
[35] Salazar, N. A.; Alvarez, C.; Orrego, C. E. Optimization of Freezing Parameters for Freeze-Drying Mango (*Mangifera Indica* L.) Slices. *Dry. Technol*. **2017**. in press. DOI: 10.1080/07373937.2017.1315431.
[36] Tasirin, S. M.; Puspasari, I.; Sahalan, A. Z.; Mokhtar, M.; Ghani, M. K. A.; Yaakob, Z. Drying of *Citrus sinensis* Peels in an Inert Fluidized Bed: Kinetics, Microbiological Activity, Vitamin C, and Limonene Determination. *Dry. Technol*. **2014**, 32, 497–508.
[37] Ghanem Romdhane, N.; Bonazzi, C.; Kechaou, N.; Mihoubi, N. B. Effect of Air-Drying Temperature on Kinetics of Quality Attributes of Lemon (*Citrus limon* Cv. Lunari) Peels. *Dry. Technol*. **2015**, 33, 1581–1589.
[38] Adams, G. D. J. Freeze-Drying of Biological Materials. *Dry. Technol*. **1991**, 9, 891–925.
[39] Chen, M. L.; Yang, D. J.; Liu, S. C. Effects of Drying Temperature on the Flavonoid, Phenolic Acid and Antioxidative Capacities of the Methanol Extract of Citrus Fruit (*Citrus sinensis* (L.) Osbeck) Peels. *Int. J. Food Sci. Tech*. **2011**, 46, 1179–1185.
[40] Lou, S. N.; Hsu, Y. S.; Ho, C. T. Flavonoid Compositions and Antioxidant Activity of Calamondin Extracts Prepared Using Different Solvents. *J. Food Drug. Anal*. **2014**, 22, 290–295.
[41] Ledesma-Escobar, C. A.; Priego-Capote, F.; Luque de Castro, M. D. Comparative Study of the Effect of Sample Pretreatment and Extraction on the Determination of Flavonoids from Lemon (*Citrus limon*). *PloS One*. **2016**, 11, DOI: 10.1371/journal.pone.0148056.
[42] Assefa, A. D.; Keum, Y. S. Effect of Extraction Solvent and Various Drying Methods on Polyphenol Content and Antioxidant Activities of Yuzu (*Citrus junos* Sieb Ex Tanaka). *J. Food Meas. Char*. **2017**, 11, 576–585.

[43] Lou, S. N.; Lai, Y. C.; Huang, J. D.; Ho, C. T.; Ferng, L. H. A.; Chang, Y. C. Drying Effect on Flavonoid Composition and Antioxidant Activity of Immature Kumquat. *Food Chem*. **2015**, 171, 356–363.
[44] Ledesma-Escobar, C. A.; Priego-Capote, F.; Luque de Castro, M. D. Effect of Sample Pretreatment on the Extraction of Lemon (*Citrus limon*) Components. *Talanta*. **2016**, 153, 386–391.
[45] Lou, S. N.; Lin, Y. S.; Hsu, Y. S.; Chiu, E. M.; Ho, C. T. Soluble and Insoluble Phenolic Compounds and Antioxidant Activity of Immature Calamondin Affected by Solvents and Heat Treatment. *Food Chem*. **2014**, 161, 246–253.
[46] Delgado-Nieblas, C. I.; Zazueta-Morales, J. J.; Ahumada-Aguilar, J. A.; Aguilar-Palazuelos, E.; Carrillo-López, A.; Jacobo-Valenzuela, N.; Telis-Romero, J. Optimization of an Air-Drying Process to Obtain a Dehydrated Naranjita (*Citrus mitis* B.) Pomace Product with High Bioactive Compounds and Antioxidant Capacity. *J. Food Process. Eng*. **2017**, 40, DOI: 10.1111/jfpe.12338.
[47] Garau, M. C.; Simal, S.; Rosselló, C.; Femenia, A. Effect of Air-Drying Temperature on Physico-Chemical Properties of Dietary Fibre and Antioxidant Capacity of Orange (*Citrus aurantium* V. Canoneta) By-Products. *Food Chem*. **2007**, 104, 1014–1024.
[48] McSweeney, M.; Seetharaman, K. State of Polyphenols in the Drying Process of Fruits and Vegetables. *Crit. Rev. Food Sci. Nutr*. **2015**, 55, 660–669.
[49] Lavelli, V.; Pompei, C.; Casadei, M. A. Quality of Nectarine and Peach Nectars as Affected by Lye-Peeling and Storage. *Food Chem*. **2009**, 115, 1291–1298.
[50] Rohn, S.; Buchner, N.; Driemel, G.; Rauser, M.; Kroh, L. W. Thermal Degradation of Onion Quercetin Glucosides under Roasting Conditions. *J. Agric. Food Chem*. **2007**, 55, 1568–1573.
[51] Rechner, A. R.; Spencer, J. P.; Kuhnle, G.; Hahn, U.; Rice-Evans, C. A. Novel Biomarkers of the Metabolism of Caffeic Acid Derivatives in Vivo. *Free Radic. Biol. Med*. **2001**, 30, 1213–1222.
[52] Bal, R.; Tope, B. B.; Sivasanker, S. Vapour Phase O-Methylation of Dihydroxy Benzenes with Methanol over Cesium-Loaded Silica, a Solid Base. *J. Mol. Catal. A: Chem*. **2002**, 181, 161–171.
[53] M'hiri, N.; Ioannou, I.; Ghoul, M.; Boudhrioua, N. M. Proximate Chemical Composition of Orange Peel and Variation of Phenols and Antioxidant Activity during Convective Air Drying. *J. New Sci*. **2015**, 9, 881–890.
[54] Ghanem Romdhane, N.; Djendoubi, N.; Bonazzi, C.; Kechaou, N.; Boudhrioua Mihoubi, N. Effect of Combined Air-Drying-Osmotic Dehydration on Kinetics of Techno-Functional Properties, Color and Total Phenol Contents of Lemon (*Citrus limon* V. Lunari) Peels. *Int. J. Food Eng*. **2016**, 12, 515–525.
[55] Alibas, I. Microwave, Vacuum, and Air Drying Characteristics of Collard Leaves. *Dry. Technol*. **2009**, 27, 1266–1273.
[56] Jaya, S.; Das, H. A Vacuum Drying Model for Mango Pulp. *Dry. Technol*. **2003**, 21, 1215–1234.
[57] Zoric, Z.; Dragovic-Uzelac, V.; Pedisic, S.; Kurtanjek, Z.; Garofulic, I. E. Kinetics of the Degradation of Anthocyanins, Phenolic Acids and Flavonols during Heat Treatments of Freeze-Dried Sour Cherry Marasca Paste. *Food Technol. Biotechnol.*. **2014**, 52, 101–108.
[58] Díaz, G. R. Z.; Martínez-Monzó, J.; Fito, P.; Chiralt, A. Modelling of Dehydration-Rehydration of Orange Slices in Combined Microwave/Air Drying. *Innov. Food Sci. Emerg. Technol*. **2003**, 4, 203–209.
[59] Ghanem, N.; Mihoubi, D.; Kechaou, N.; Mihoubi, N. B. Microwave Dehydration of Three Citrus Peel Cultivars: Effect on Water and Oil Retention Capacities, Color, Shrinkage and Total Phenols Content. *Ind. Crops Prod*. **2012**, 40, 167–177.
[60] Bejar, K. A.; Kechaou, N.; Boudhrioua Mihoubi, N. Effect of Microwave Treatment on Physical and Functional Properties of Orange (*Citrus sinensis*) Peel and Leaves. *J. Food Process. Technol*. **2011**, 2, 109–116.

[61] Riadh, M. H.; Ahmad, S. A. B.; Marhaban, M. H.; Soh, A. C. Infrared Heating in Food Drying: An Overview. *Dry. Technol.* **2015**, 33, 322–335.
[62] Nowak, D.; Lewicki, P. P. Infrared Drying of Apple Slices. *Innov. Food Sci. Emerg. Technol.* **2004**, 5, 353–360.
[63] Luthria, D. L. Influence of Experimental Conditions on the Extraction of Phenolic Compounds from Parsley (Petroselinum Crispum) Flakes Using a Pressurized Liquid Extractor. *Food Chem.* **2008**, 107, 745–752.
[64] Xu, G.; Ye, X.; Chen, J.; Liu, D. Effect of Heat Treatment on the Phenolic Compounds and Antioxidant Capacity of Citrus Peel Extract. *J. Agric. Food Chem.* **2007**, 55, 330–335.
[65] Ma, Y. Q.; Ye, X. Q.; Fang, Z. X.; Chen, J. C.; Xu, G. H.; Liu, D. H. Phenolic Compounds and Antioxidant Activity of Extracts from Ultrasonic Treatment of Satsuma Mandarin (*Citrus unshiu* Marc.). *Peels. J. Agric. Food Chem.* **2008**, 56, 5682–5690.
[66] Bilbao, M. L. M.; Andrés-Lacueva, C.; Jáuregui, O.; Lamuela-Raventós, R. M. Determination of Flavonoids in a Citrus Fruit Extract by LC–DAD and LC–MS. *Food Chem.* **2007**, 101, 1742–1747.
[67] Lodish, H.; Berk, A.; Zipurski, S. L.; Matsudaira, P.; Baltimore, D.; Darnell, J. *Molecular Cell Biology 4th Edition*; National Center for Biotechnology InformationÕs Bookshelf, New York, NY, **2000**.
[68] Hayat, K.; Hussain, S.; Abbas, S.; Farooq, U.; Ding, B.; Xia, S.; Jia, C.; Zhang, X.; Xia, W. Optimized Microwave-Assisted Extraction of Phenolic Acids from Citrus Mandarin Peels and Evaluation of Antioxidant Activity *in Vitro*. *Sep. Purif. Technol.* **2009**, 70, 63–70.
[69] Nayak, B.; Dahmoune, F.; Moussi, K.; Remini, H.; Dairi, S.; Aoun, O.; Khodir, M. Comparison of Microwave, Ultrasound and Accelerated-Assisted Solvent Extraction for Recovery of Polyphenols from *Citrus sinensis* Peels. *Food Chem.* **2015**, 187, 507–516.
[70] Kaufmann, B.; Christen, P.; Veuthey, J. L. Parameters Affecting Microwave-Assisted Extraction of Withanolides. *Phytochem. Anal.* **2001**, 12, 327–331.
[71] Sharma, K.; Ko, E. Y.; Assefa, A. D.; Ha, S.; Nile, S. H.; Lee, E. T.; Park, S. W. Temperature-Dependent Studies on the Total Phenolics, Flavonoids, Antioxidant Activities, and Sugar Content in Six Onion Varieties. *J. Food Drug Anal.* **2015**, 23, 243–252.
[72] Perkins-Veazie, P.; Collins, J. K.; Howard, L. Blueberry Fruit Response to Postharvest Application of Ultraviolet Radiation. *Postharvest Biol. Technol.* **2008**, 47, 280–285.
[73] Liu, C. H.; Cai, L. Y.; Lu, X. Y.; Han, X. X.; Ying, T. J. Effect of Postharvest UV-C Irradiation on Phenolic Compound Content and Antioxidant Activity of Tomato Fruit during Storage. *J. Integr. Agr.* **2012**, 11, 159–165.
[74] Young, A. R. Acute Effects of UVR on Human Eyes and Skin. *Prog. Biophys. Mol. Biol.* **2006**, 92, 80–85.
[75] Katiyar, S. K.; Pal, H. C.; Prasad, R. Dietary Proanthocyanidins Prevent Ultraviolet Radiation-Induced Non-Melanoma Skin Cancer through Enhanced Repair of Damaged DNA-dependent Activation of Immune Sensitivity. *Semin. Cancer Biol.* **2017**, 46, 138–145.
[76] Lung, H. M.; Cheng, Y. C.; Chang, Y. H.; Huang, H. W.; Yang, B. B.; Wang, C. Y. Microbial Decontamination of Food by Electron Beam Irradiation. *Trends Food Sci. Technol.* **2015**, 44, 66–78.
[77] Fernandes, Â.; Antonio, A. L.; Oliveira, M. B. P. P.; Martins, A.; Ferreira, I. C. F. R. Effect of Gamma and Electron Beam Irradiation on the Physico-Chemical and Nutritional Properties of Mushrooms: A Review. *Food Chem.* **2012**, 135, 641–650.
[78] Goulas, V.; Manganaris, G. A. Exploring the Phytochemical Content and the Antioxidant Potential of Citrus Fruits Grown in Cyprus. *Food Chem.* **2012**, 131, 39–47.
[79] Bermejo, A.; Llosá, M. J.; Cano, A. Analysis of Bioactive Compounds in Seven Citrus Cultivars. *Food Sci. Technol. Int.* **2011**, 17, 55–62.
[80] Sun, Y.; Wang, J.; Gu, S.; Liu, Z.; Zhang, Y.; Zhang, X. Simultaneous Determination of Flavonoids in Different Parts of Citrus Reticulata 'Chachi' Fruit by High Performance Liquid Chromatography – Photodiode Array Detection. *Molecules.* **2010**, 15, 5378–5388.
[81] Wang, Y. C.; Chuang, Y. C.; Hsu, H. W. The Flavonoid, Carotenoid and Pectin Content in Peels of Citrus Cultivated in Taiwan. *Food Chem.* **2008**, 106, 277–284.

[82] Ramful, D.; Bahorun, T.; Bourdon, E.; Tarnus, E.; Aruoma, O. I. Bioactive Phenolics and Antioxidant Propensity of Flavedo Extracts of Mauritian Citrus Fruits: Potential Prophylactic Ingredients for Functional Foods Application. *Toxicology*. **2010**, 278, 75–87.
[83] Bocco, A.; Cuvelier, M. E.; Richard, H.; Berset, C. Antioxidant Activity and Phenolic Composition of Citrus Peel and Seed Extracts. *J. Agric. Food Chem*. **1998**, 46, 2123–2129.
[84] Del Río, J. A.; Fuster, M. D.; Gómez, P.; Porras, I.; García-Lidón, A.; Ortuño, A. *Citrus limon*: A Source of Flavonoids of Pharmaceutical Interest. *Food Chem*. **2004**, 84, 457–461.
[85] Xu, G. H.; Chen, J.; Liu, D. H.; Zhang, Y. H.; Jiang, P.; Ye, X. Q. Minerals, Phenolic Compounds, and Antioxidant Capacity of Citrus Peel Extract by Hot Water. *J. Food Sci*. **2008**, 73, C11–C18.
[86] Wang, Y. C.; Chuang, Y. C.; Ku, Y. H. Quantitation of Bioactive Compounds in Citrus Fruits Cultivated in Taiwan. *Food Chem*. **2007**, 102, 1163–1171.
[87] Ma, Y. Q.; Chen, J. C.; Liu, D. H.; Ye, X. Q. Simultaneous Extraction of Phenolic Compounds of Citrus Peel Extracts: Effect of Ultrasound. *Ultrason. Sonochem*. **2009**, 16, 57–62.
[88] Peleg, H.; Naim, M.; Rouseff, R. L.; Zehavi, U. Distribution of Bound and Free Phenolic Acids in Oranges (*Citrus sinensis*) and Grapefruits (*Citrus paradisi*). *J. Sci. Food Agr*. **1991**, 57, 417–426.
[89] Wojdyło, A.; Figiel, A.; Lech, K.; Nowicka, P.; Oszmiański, J. Effect of Convective and Vacuum–Microwave Drying on the Bioactive Compounds, Color, and Antioxidant Capacity of Sour Cherries. *Food Bioproc. Tech*. **2014**, 7, 829–841.
[90] Talens, C.; Arboleya, J. C.; Castro-Giraldez, M.; Fito, P. J. Effect of Microwave Power Coupled with Hot Air Drying on Process Efficiency and Physico-Chemical Properties of a New Dietary Fibre Ingredient Obtained from Orange Peel. *LWT-Food Sci. Technol*. **2017**, 77, 110–118.