

## Review

## Flavour chemistry of dehydrated exotic fruits

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### Abstract

Dehydrated fruit pieces and purées are commonly used in many food formulations and toppings for food products like yogurts, ice creams, and cereals. However, one of the biggest problems in fruit dehydration is the prevention of the development of unpleasant off-flavour that can negatively impact on the sensory quality of the final dried fruit products. In recent years, fruit dehydration has been conducted using either thermal or non-thermal drying techniques. Therefore, the present review examines how the different dehydration technologies, namely conventional hot air-drying, ultrasonic-assisted hot air-drying, sun-drying, spray-drying, Refractance Window™ drying, cast-tape drying, thin-layer catalytic far-infrared radiation drying, withering, freeze-drying, microwave-drying, and osmotic dehydration impact on the volatile constituents of the final dried fruit products. Drying processes result in noticeable losses/reduction of several impact odorants. Moreover, some compounds are produced either via: (1) hydrolysis of relevant glycosides under high temperatures, or (2) thermal degradation of volatile and non-volatile precursors as well as oxidation and Maillard reactions which result in the production of heterocyclics, and saturated and unsaturated aldehydes. Of significance is the Refractance Window™ drying which exhibits high retention potential (~ 90%) of volatile compounds present in fresh fruits. Refractance Window™ drying technology ensures rapid drying of food products at very low temperature.

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### Introduction

Drying is a process whereby moisture is vaporised from the food's surface. Drying is quite unique for its noteworthy extension of food shelf stability. Drying has also been employed in the production of fruit pieces (Martynenko and Janaszek, 2014). Fruit dehydration is known to increase the cohesiveness of the final product (Martynenko and Janaszek, 2014). Moreover, drying can induce cell shrinkage which could result in poor rehydration of the dried fruit (Martynenko and Janaszek, 2014). The impact of drying on the flavour qualities of several dried fruits such as apple food products (Li *et al.*, 2010; Mothibe *et al.*, 2014; Tsuruta *et al.*, 2015), white dehydrated grapes (Rolle *et al.*, 2012), air-dried raisins (Wang *et al.*, 2015), dried mango (*Mangifera indica*; Bonneau *et al.*, 2016), dehydrated pear (*Pyrus*

*communis* L.; Komes *et al.*, 2007), dried white figs (*Ficus carica* L.; Mujić *et al.*, 2014), and dried lulo fruits (*Solanum quitoense* Lam; Forero *et al.*, 2015) has been reported. Upon drying, moisture is lost from the outer surface of the fruit, and with further drying, cell shrinkage sets in. Usually, the shrinkage will normally start from the fruit's surface and progress inward. Studies have validated similar trend for the loss of volatile compounds during drying of fruits (Goubet *et al.*, 1998; Conte *et al.*, 2019). In addition, the properties of the flavour compounds such as functional groups, polarity, relative volatility, and molecular weight could affect the retention of volatile compounds in the product. One unique problem of fruit dehydration is the development of unpleasant off-flavour in the dried fruit (Osorio *et al.*, 2011). To solve this problem, researchers have adopted the non-thermal drying techniques in drying fruits. Some of

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the noteworthy non-thermal drying methods which are currently being used are lyophilisation (Ceballos *et al.*, 2012), osmotic dehydration (Osorio *et al.*, 2007), and refractance window system (Abonyi *et al.*, 2002). Therefore, the present review examines how the different dehydration technologies impact on the volatile constituents of the final dried fruit products.

#### *Analysis of volatile compounds in dehydrated fruits*

Various trapping technologies have been used to collect volatile compounds from fruit samples, either directly or after concentration, as in head-space techniques. Some examples of these methods are the dynamic head-space or purge-and-trap technique. These methods have successfully been employed with dehydrated cherry tomatoes (Heredia *et al.*, 2012) and dried carrots (*Daucus carota* L. ssp. *sativus* var. *atrorubens* Alef.; Keskin *et al.*, 2021; Guclu *et al.*, 2021). Similarly, solvent assisted flavour evaporation (SAFE) and head-space solid phase micro-extraction (HS-SPME) are examples of other trapping methods regularly used in the extraction of volatile compounds from food matrices. For example, Bonneau *et al.* (2016) and Forero *et al.* (2015) employed SAFE to extract volatile compounds from dried mangoes and dried lulo fruits, respectively. On the other hand, HS-SPME has been employed in dried fruits like tomatoes (Rajkumar *et al.*, 2021), figs (*Ficus carica* L.; Russo *et al.*, 2017), and apple slices (Conte *et al.*, 2019). Less frequently used extraction methods are the stir bar sorptive extraction (SBSE) and distillation under reduced pressure. However, two recent studies reported on the effectiveness of both techniques. For example, SBSE was used in the spray-dried soursop (*Annona muncata*; Neta *et al.*, 2019), and distillation under reduced pressure was employed in dried mulberry (*Morus alba* L.; Hwang and Kim, 2020).

Extraction is usually followed by volatiles' analysis, and this is often carried out with the aid of versatile instruments such as gas chromatography with flame ionisation detection (GC-FID), gas chromatography-olfactometry (GC-O), gas chromatography-mass spectrometry (GC-MS), and the comprehensive two-dimensional gas chromatography-mass spectrometry (GCx GC-MS) (Raice *et al.*, 2015; Allamy *et al.*, 2018; Neta *et al.*, 2019). These different techniques provide excellent sensitivity and results.

#### *Effect of different dehydration techniques on aroma formation and losses in dehydrated fruits*

##### *Conventional hot air-drying of fruits Apple (*Malus domestica* Borkh.)*

Slices of apple cultivar Golden Delicious (10.0 ± 0.2 mm thick × 20.0 ± 0.3 mm diameter) were dried using conventional hot air-drying unit, and was reported by Conte *et al.* (2019). In that study, three drying methods were employed. The first method involved microwave drying at 35, 55, and 65°C. The second drying method involved the use of hot air drying at 55 and 65°C, respectively. The third method was a combination of both hot air- and microwave-drying at 65°C. Fifty volatile compounds of different groups of compounds namely esters, aldehydes, alcohols, and acids were reported in the fresh apple fruit. In that study, esters were the main volatile compounds identified. Some of the esters were butylacetate, 2-methyl-1-butyl acetate, and hexyl acetate. Another prominent class of compounds identified were the aldehydes with hexanal and 2-hexenal being the main aldehydes. The application of different drying methods significantly affected the overall volatile compounds concentration in the dried apple samples. For example, the hot air-drying method produced more pronounced reductions in the levels of the esters and aldehydes in the dried apples as compared to other drying methods such as microwave or the combination of hot air- and microwave-drying, respectively. In that study, the probable losses in the VOCs could be associated with stripping. On the other hand, the formation of new VOCs was mainly due to oxidation, thermal degradation, and the conversion of non-volatile precursors to new flavour components.

Previous study on the formation of flavour compounds by the reaction of amino acid and carbonyl compounds under mild conditions has shown that thermal degradation of volatile and non-volatile precursors, as well as oxidation and Maillard reactions often result in the production of heterocyclics, as well as aldehydes (*i.e.*, saturated and unsaturated) (Pripis-Nicolau *et al.*, 2000). Most times, some of these aldehydes cause off-flavour. For example, while 5-methyl-2-furfural, furan-2-carbaldehyde, benzaldehyde, and 2,4-decadienal were found in the dried apples, they were absent in the fresh apples (Conte *et al.*, 2019).

*Carrot (Daucus carota L.)*

The drying of shredded fresh black carrot sample (250 g) at 60°C was reported by Keskin *et al.* (2021), which generated 32 volatile compounds comprising of alcohols, aldehydes, acids, esters, and terpenes. Drying of the black carrots significantly reduced the quantities of the volatiles identified in the dried carrots. For instance, drying produced almost 39% reduction in the total concentration of volatiles in the dried carrots. While the fresh black carrots recorded a total volatile concentration of 40,699 µg kg<sup>-1</sup>, the dried carrots had a much lower concentration (15,988 µg kg<sup>-1</sup>). The terpenes were the predominant group of volatiles in both the fresh and the dried black carrots.

Kebede *et al.* (2014) have earlier reported the presence of high amounts of terpenes in other carrots. It is worthy of note that there were significant decreases in the levels of monoterpenes found in the dried carrots. For example, the β-myrcene, terpinolene, α-pinene, and limonene were noticeably reduced in the dried black carrots (Keskin *et al.*, 2021). The observed losses could be a result of their high partition coefficient. Earlier study has shown the high susceptibility of these compounds to losses via evaporation (Bonneau *et al.*, 2016). Other compounds affected substantially by drying in the black carrots were alcohols and esters (isobornyl acetate and methyl palmitate). 2-furanmethanol (furfuryl alcohol) was only found in the dried carrot samples (Keskin *et al.*, 2021).

*Fig (Ficus carica L. cv. Dottato)*

The drying of whole fig samples in a pilot plant cabinet dryer at 60°C and 25% (wet basis) was reported by Russo *et al.* (2017), which generated 42 volatile compounds with the aldehydes accounting for approximately 80% of the compounds. Of these, benzaldehyde and hexanal were the most abundant aldehydes identified in the dried figs. Conversely, ethyl alcohol, 1-dodecanol, 1-octen-3-ol, 3-methylbutanal, pentanal, heptanal, nonanal, octanal, and ethyl acetate occurred in lower concentrations. While drying of figs resulted in significant increases in furans and terpenes in the dried fruits, it however, resulted in significant decreases in alcohols and esters. In addition, aldehydes and ketones showed noticeable changes after drying; for example, there was a remarkable decrease in the concentration of hexanal while a corresponding increase in the concentration of benzaldehyde was reported.

Moreover, compounds such as 2-heptanone, 2-octanone, 2-nonanone, D-limonene, 2-pentylfuran, nonanal, decanal, and (*E*)-2-decenal recorded significant increases. This is probably due to oxidation or the effect of drying (Russo *et al.*, 2017).

*Lime [Citrus aurantifolia (Christm.) Swingle]*

The drying of lime fruits was reported by Ramesh Yadav *et al.* (2004), which generated 32 volatile compounds. Monoterpene hydrocarbons accounted for approximately 83% of the classes of compounds reported in both fresh and dried lime fruits. This was followed by the alcohols (18%). Other classes of compounds identified with appreciable abundance in the lime fruit were the sesquiterpenes, aldehydes, and esters, respectively. The major compound identified in the lime fruits was the cyclic monoterpene and limonene, and other volatiles with appreciable abundances were the β-pinene, γ-terpinene, nerolidol, and α-terpineol. In addition, the following compounds namely neryl acetate, geranyl acetate, neral, geranial, dodecanal, and tetradecanal were also identified in both fresh and dried fruits. It is worthy of note that drying did not significantly alter the quantities of volatiles in both fresh and dried lime fruits.

*Mulberry (Morus alba L.)*

The drying of mulberry fruits at either 50 or 60°C was reported by Hwang and Kim (2020), which generated 30 volatile compounds. Of these, 12 volatile compounds were found in both fresh and dried fruits, respectively. Some of these compounds were benzaldehyde, ethyl acetate, 1,3-cyclohexadiene, 2,4-dimethylheptane, 4-methyloctane, cyclopentane, carboxaldehyde, cyclohexene oxide, 2-cyclohexen-1-one, phenol, and nonadecane. These compounds occurred in different concentrations in either mulberry samples dried at either 50 or 60°C. For example, ethyl acetate recorded significantly ( $p < 0.05$ ) higher concentration in mulberry fruits dried at 50°C than those dried at 60°C. This was followed by the fresh sweet 2-cyclohexen-1-one. Another compounds identified in the dried mulberry fruits is 2-cyclohexen-1-ol (Jo *et al.*, 2013).

*Ultrasonic-assisted hot air-drying (HUD) of fruits**Lulo (Solanum quitoense Lam.) fruit*

The ultrasonic-assisted hot air-drying of homogenised lulo fruit pulp with maltodextrin was reported by Forero *et al.* (2015), which generated

only 16 volatile compounds. The major classes of compounds were esters (methyl benzoate, methyl butanoate, ethyl butanoate, ethyl hexanoate, and methyl hexanoate), aldehydes (hexanal, (Z)-3-hexenal, (E)-2-hexenal), acids (acetic acid and butanoic acid), and alcohols (methyl-2-butanol, (Z)-3-hexen-1-ol), (Z)-3-hexenal, and methyl hexanoate (fruity)). Most of the C6 compounds such (Z)-3-hexenal, (E)-2-hexenal, and the corresponding alcohol and ester derivatives are often referred to green volatiles because of their leafy-green aroma nuance. Most of the green volatiles are biosynthesised from polyunsaturated fatty acids in the thylakoid membranes by a series of enzymes (Kunishima *et al.*, 2016). In addition, (E)-2-hexenal has been reported as a product of (Z)-3-hexenal isomerisation in the biosynthesis pathway, although, the enzyme responsible has not been identified (Kunishima *et al.*, 2016). Moreover, drying resulted in the losses of acetic acid, (Z)-3-hexenal, (Z)-3-hexen-1-ol, and butanoic acid. In contrast, the drying process had only slight effect on the esters.

#### *Grape (Vitis vinifera L.)*

The air-drying of seedless grape cultivars namely Thompson Seedless, Flame Seedless, and Crimson Seedless in ventilated and lucifugal adobe houses ( $3 \times 4 \times 6 - 8 \text{ m}^3$ ) was reported by Fang *et al.* (2010), which generated 73 volatile compounds of which alcohols, aldehydes, and terpenoids accounted for the majority of the classes of compounds. Of these, 39 compounds were found in the free and bound forms of the raisins (Table 1). On the other hand, 34 volatile compounds were reported only in the free form (Wang *et al.*, 2015). In addition, 13 aldehydes which were identified in that study only occurred in the free form in the raisins. Esters were also found in the raisins only in the free forms. The only exception to this was methyl salicylate. Methyl salicylate was found in the bound-form in Muscat grape (Baek and Cadwallader, 1999). In addition, compounds like lilac alcohol, neral, hexadecanoic acid, (E)- $\beta$ -ocimene, and diethyl succinate were identified for the first time in the raisins. Only three pyrazines (2-ethyl-6-methyl pyrazine, 3-ethyl-2,5-dimethyl pyrazine, and 2,6-diethylpyrazine) were identified in the raisins. The furans were only identified in the free form with furfural having the highest concentration.

Most of the terpenes identified in the raisins were identified for the first time. The only exceptions

to this were the geranylacetone and  $\alpha$ -terpineol (Wang *et al.*, 2015). In that study, nerol, geraniol, and geranic acid which occurred in the free form generated the greatest values (149 to  $237 \mu\text{g L}^{-1}$ ), respectively. On the other hand, higher concentrations were obtained for the bound forms of  $\beta$ -damascenone, geraniol, geranic acid, neral, and nerol (Wang *et al.*, 2015).

#### *Innovative drying technologies*

##### *Cast-tape drying*

The drying of pineapple (*Ananas comosus* (L.) Merrill) pulp-cassava suspension using the cast-tape method (Figure 1) was reported by Simao *et al.* (2021). This significantly reduced the number of volatile compounds identified in the fresh samples. The major volatile compounds found in the dried samples were hexadecane, 2,5-dimethyl-4-hydroxy-3(2H)-furanone, bis (2-ethylhexyl) adipate, and acetic acid. These results constituted a reduction of about 45% of the initial volatile compounds in the fresh samples. In that study, 2,5-dimethyl-4-hydroxy-3(2H)-furanone and acetic acid were newly generated compounds in the dried samples. Cast-tape drying resulted in the losses of ethanol, 2-phenylethanol, ethyl acetate, isoamyl acetate, and phenethyl acetate, respectively.

##### *Refractance Window<sup>TM</sup> drying*

The drying of "Bocadillo banana" (*Musa acuminata* Colla) slices (2.3 cm diameter) using drying window refractance (RW) (Figure 2) at 70, 80, and 90°C was reported by Ormaza *et al.* (2016), which generated 30 volatile compounds. Of these, aldehydes, alcohols, acids, esters, and ketones were the predominant groups of compounds identified in the dried banana slices. The major volatile compounds were 2-pentanone, 2-methyl-1-propanol, 2-pentanol, 3-methyl-1-butanol, isoamyl butyrate, 3-hydroxy-2-butanal, methyl isovalerate, and acetic acid. The window refractance retained on average 80% of the volatile compounds. The treatment at 80°C was better as it retained the highest percentage (91%) of the volatile compounds. The reason for this observation is not farfetched since RWD has been known to retain aroma and other qualities of dried food products (Bernaert *et al.*, 2019). The beauty of RWD technology is that it is an efficient and rapid drying method that dries product at very low temperature.

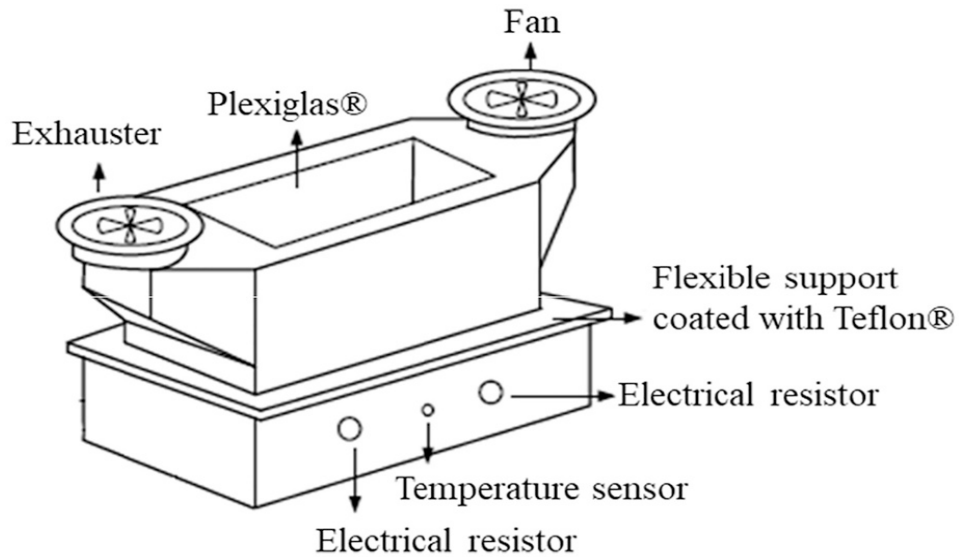
**Table 1.** The concentration ( $\mu\text{g.L}^{-1}$ )<sup>A</sup> of free and bound volatile compounds in the three raisin varieties.

No	RI	Compound	Ion ( $m/z$ ) <sup>B</sup>	ID <sup>C</sup>	Free compound ( $\mu\text{g.L}^{-1}$ )			Bound compound ( $\mu\text{g.L}^{-1}$ )		
					TSR	FSR	CSR	TSR	FSR	CSR
<b>Alcohol</b>										
1	1203	3-methyl-1-butanol	55	1	0.57 ± 0.08 <sup>a</sup>	0.81 ± 0.03 <sup>b</sup>	0.52 ± 0.04 <sup>a</sup>	462 ± 21 <sup>ab</sup>	383 ± 79 <sup>a</sup>	714 ± 87 <sup>b</sup>
2	1205	1-pentanol	42	2	5.40 ± 0.31	3.67 ± 0.52	5.15 ± 0.51	13.5 ± 0.8 <sup>ab</sup>	11.3 ± 2.6 <sup>a</sup>	20.7 ± 2.4 <sup>b</sup>
3	1317	3-methyl-2-buten-1-ol	71	2	nd	0.28 ± 0.07	nd	102 ± 3 <sup>a</sup>	157 ± 1 <sup>b</sup>	87.5 ± 5.2 <sup>a</sup>
4	1349	1-hexanol	56	2	70.8 ± 5.1 <sup>b</sup>	57.0 ± 9.7 <sup>ab</sup>	43.3 ± 0.2 <sup>a</sup>	tr	tr	75.4 ± 11.9
5	1453	1-heptanol	70	2	2.60 ± 0.01 <sup>b</sup>	2.51 ± 0.03 <sup>ab</sup>	2.47 ± 0.03 <sup>a</sup>	8.79 ± 0.46	9.00 ± 0.04	10.4 ± 1.3
6	1458	6-methyl-5-hepten-2-ol	95	2	0.19 ± 0.03 <sup>a</sup>	0.22 ± 0.01 <sup>a</sup>	0.32 ± 0.00 <sup>b</sup>	0.24 ± 0.00 <sup>ab</sup>	0.17 ± 0.01 <sup>a</sup>	0.33 ± 0.04 <sup>b</sup>
7	1449	1-octen-3-ol	57	1	21.0 ± 1.6 <sup>b</sup>	8.5 ± 0.1 <sup>a</sup>	26.8 ± 0.9 <sup>c</sup>	2.86 ± 0.00 <sup>b</sup>	2.20 ± 0.22 <sup>a</sup>	4.05 ± 0.16 <sup>c</sup>
8	1487	2-ethyl-1-hexanol	57	1	0.74 ± 0.16	0.53 ± 0.06	0.48 ± 0.11	3.57 ± 0.05 <sup>b</sup>	tr <sup>a</sup>	10.4 ± 4.8 <sup>c</sup>
9	1555	1-octanol	56	1	2.70 ± 0.00 <sup>a</sup>	2.71 ± 0.00 <sup>a</sup>	3.27 ± 0.13 <sup>b</sup>	5.12 ± 0.40 <sup>a</sup>	6.70 ± 0.17 <sup>b</sup>	7.06 ± 0.02 <sup>b</sup>
10	1614	(E)-2-octen-1-ol	57	2	7.51 ± 0.06 <sup>b</sup>	2.59 ± 0.17 <sup>a</sup>	6.85 ± 0.96 <sup>b</sup>	0.37 ± 0.01 <sup>a</sup>	0.35 ± 0.01 <sup>a</sup>	0.45 ± 0.00 <sup>b</sup>
11	1657	1-nonanol	56	1	0.08 ± 0.01 <sup>a</sup>	0.10 ± 0.01 <sup>a</sup>	0.19 ± 0.02 <sup>b</sup>	7.46 ± 0.28 <sup>a</sup>	9.50 ± 0.20 <sup>b</sup>	7.47 ± 0.16 <sup>a</sup>
12	1879	benzyl alcohol	79	1	4.91 ± 0.37 <sup>a</sup>	36.30 ± 2.01 <sup>b</sup>	4.33 ± 0.12 <sup>a</sup>	1459 ± 140 <sup>b</sup>	2931 ± 343 <sup>c</sup>	501 ± 33 <sup>a</sup>
13	1914	2-phenylethanol	91	1	10.1 ± 0.7 <sup>a</sup>	33.4 ± 5.6 <sup>b</sup>	30.7 ± 0.1 <sup>b</sup>	49.2 ± 2.2	41.3 ± 11.7	63.9 ± 2.8
<b>Acid</b>										
14	1571	2-methylpropanoic acid	43	1	66.9 ± 5.0	90.8 ± 9.2	90.1 ± 6.0	nd	nd	nd
15	1740	pentanoic acid	60	2	47.5 ± 1.1 <sup>a</sup>	43.3 ± 1.7 <sup>a</sup>	64.6 ± 2.9 <sup>b</sup>	nd	nd	nd
16	1847	hexanoic acid	60	1	403 ± 41 <sup>a</sup>	336 ± 75 <sup>a</sup>	662 ± 38 <sup>b</sup>	tr	tr	tr
17	1950	2-ethylhexanoic acid	88	2	122 ± 0	122 ± 0.1	122 ± 0.4	71.2 ± 0.0 <sup>a</sup>	75.1 ± 0.5 <sup>b</sup>	71.6 ± 0.0 <sup>a</sup>
18	1953	heptanoic acid	60	1	56.1 ± 3.0	59.9 ± 6.4	78.2 ± 13.6	41.6 ± 1.2 <sup>a</sup>	59.0 ± 4.8 <sup>b</sup>	51.3 ± 2.9 <sup>ab</sup>
19	2060	octanoic acid	60	1	133 ± 2	139 ± 1	137 ± 2	80.0 ± 0.7	91.96 ± 5.21	85.29 ± 0.98
20	2166	nonanoic acid	60	2	133 ± 2	139 ± 1	137 ± 2	80.0 ± 0.7	91.96 ± 5.21	85.29 ± 0.98
21	2219	<i>n</i> -decanoic acid	60	2	125 ± 2	128 ± 0.4	126 ± 1	265 ± 0.1 <sup>a</sup>	275 ± 0.4 <sup>b</sup>	271 ± 2 <sup>b</sup>
22	2484	dodecanoic acid	73	2	123 ± 0.2 <sup>a</sup>	126 ± 0.1 <sup>b</sup>	123 ± 0.04 <sup>a</sup>	3.97 ± 1.13	5.13 ± 0.33	5.56 ± 0.21
23	2698	tetradecanoic acid	73	2	122 ± 0.1 <sup>a</sup>	123 ± 0.1	122 ± 0.05	4.64 ± 1.34	5.59 ± 0.09	7.51 ± 0.24
24	2910	hexadecanoic acid	43	2	nd	nd	121 ± 0.3	tr	tr	tr

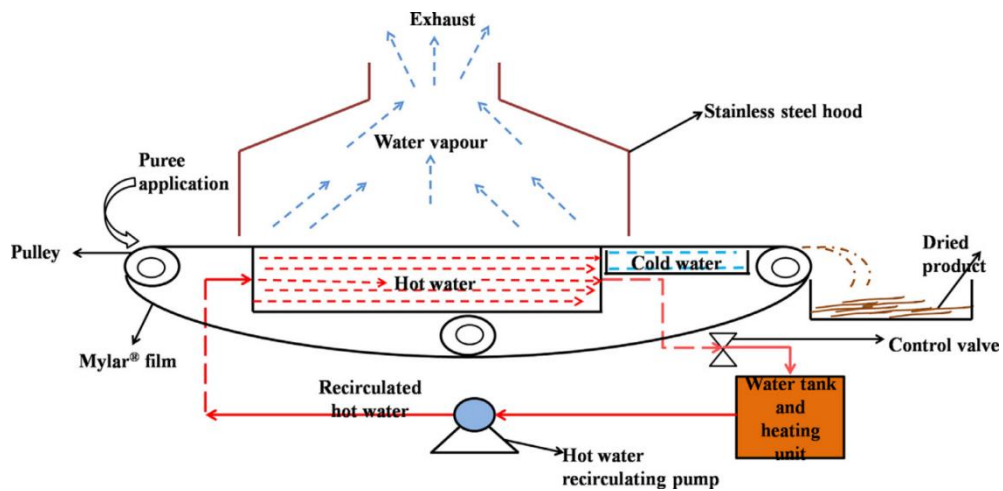
<b>Aldehyde</b>												
25	975	pentanal	44	2	10.1 ± 0.6 <sup>b</sup>	6.7 ± 1.2 <sup>a</sup>	14.4 ± 0.1 <sup>c</sup>	nd	nd	nd	nd	nd
26	1066	hexanal	44	1	49.9 ± 2.6 <sup>c</sup>	28.1 ± 2.4 <sup>b</sup>	8.79 ± 0.91 <sup>a</sup>	nd	nd	nd	nd	nd
27	1178	heptanal	44	2	7.34 ± 0.99	5.98 ± 0.31	12.5 ± 2.9	nd	nd	nd	nd	nd
28	1217	(E)-2-hexenal	41	1	30.0 ± 7.2 <sup>b</sup>	16.6 ± 1.3 <sup>a</sup>	30.5 ± 3.8 <sup>b</sup>	nd	nd	nd	nd	nd
29	1292	octanal	43	2	42.3 ± 13.2	54.5 ± 1.6	51.8 ± 2.8	nd	nd	nd	nd	nd
30	1325	(E)-2-heptenal	41	2	21.8 ± 0.0 <sup>b</sup>	tr <sup>a</sup>	57.7 ± 4.1 <sup>c</sup>	nd	nd	nd	nd	nd
31	1393	nonanal	57	1	tr	7.06 ± 2.11	5.35 ± 0.64	nd	nd	nd	nd	nd
32	1432	(E)-2-octenal	41	2	tr	tr	tr	nd	nd	nd	nd	nd
34	1530	benzaldehyde	77	1	7.29 ± 0.53 <sup>a</sup>	11.3 ± 0.4 <sup>b</sup>	8.14 ± 0.56 <sup>a</sup>	nd	nd	nd	nd	nd
35	1539	(E)-2-nonenal	43	2	nd	nd	tr	nd	nd	nd	nd	nd
36	1650	benzeneacetaldehyde	91	1	33.3 ± 2.7 <sup>c</sup>	10.9 ± 2.2 <sup>a</sup>	22.8 ± 1.4 <sup>b</sup>	nd	nd	nd	nd	nd
37	1705	(E,E)-2,4-nonadienal	81	2	tr	tr	tr	nd	nd	nd	nd	nd
<b>Ester</b>												
38	885	ethyl acetate	43	1	395 ± 10 <sup>b</sup>	420 ± 85 <sup>b</sup>	97 ± 1 <sup>a</sup>	nd	nd	nd	nd	nd
39	1227	ethyl hexanoate	88	2	6.05 ± 0.78	6.43 ± 0.53	5.86 ± 0.25	nd	nd	nd	nd	nd
40	1432	ethyl octanoate	88	2	1.26 ± 0.08	1.41 ± 0.06	1.46 ± 0.05	nd	nd	nd	nd	nd
41	1636	butyrolactone	42	2	5.05 ± 0.61	6.24 ± 3.63	nd	nd	nd	nd	nd	nd
42	1677	diethyl succinate	101	1	0.60 ± 0.07 <sup>b</sup>	0.69 ± 0.03 <sup>b</sup>	0.16 ± 0.04 <sup>a</sup>	nd	nd	nd	nd	nd
43	1734	benzyl acetate	108	2	1.02 ± 0.00 <sup>a</sup>	1.16 ± 0.00 <sup>b</sup>	1.01 ± 0.00 <sup>a</sup>	nd	nd	nd	nd	nd
44	1782	methyl salicylate	120	1	33.5 ± 4.0 <sup>a</sup>	794 ± 46 <sup>b</sup>	48.6 ± 8.1 <sup>a</sup>	10.2 ± 0.2 <sup>b</sup>	12.5 ± 0.6 <sup>c</sup>	6.0 ± 0.1 <sup>a</sup>	6.0 ± 0.1 <sup>a</sup>	6.0 ± 0.1 <sup>a</sup>
45	1820	phenethyl acetate	104	2	1.04 ± 0.00 <sup>a</sup>	1.12 ± 0.00 <sup>c</sup>	1.06 ± 0.00 <sup>b</sup>	nd	nd	nd	nd	nd
46	2200	ethyl hexadecanoate	88	2	0.15 ± 0.02	nd	0.27 ± 0.01	nd	nd	nd	nd	nd
47	2035	γ-nonalactone	85	2	17.1 ± 0.5 <sup>a</sup>	26.6 ± 3.2 <sup>b</sup>	26.6 ± 3.2 <sup>b</sup>	nd	nd	nd	nd	nd
<b>Ketone</b>												
48	1167	2,6-dimethyl-4-heptanone	57	2	1.65 ± 0.00	1.95 ± 0.18	1.91 ± 0.17	nd	nd	nd	nd	nd
49	1289	acetoin	45	1	20.9 ± 4.0	29.1 ± 0.2	15.8 ± 14.8	1423 ± 129	1347 ± 139	1922 ± 153	1922 ± 153	1922 ± 153
50	1337	6-methyl-5-hepten-2-one	43	1	2.49 ± 0.74	1.73 ± 0.00	3.23 ± 0.38	nd	nd	nd	nd	nd
51	1596	6-methyl-3,5-heptadiene-2-one	109	2	2.66 ± 0.09 <sup>b</sup>	1.59 ± 0.03 <sup>a</sup>	7.07 ± 0.20 <sup>c</sup>	nd	nd	nd	nd	nd

<b>Terpenoid</b>										
52	1252	(E)- $\beta$ -ocimene	93	2	nd	16.9 $\pm$ 0.1	nd	2.13 $\pm$ 0.45 <sup>a</sup>	2.87 $\pm$ 0.19 <sup>ab</sup>	3.46 $\pm$ 0.05 <sup>b</sup>
53	1267	<i>p</i> -cymene	119	1	15.7 $\pm$ 0.1 <sup>a</sup>	16.1 $\pm$ 0.1 <sup>b</sup>	15.6 $\pm$ 0.1 <sup>a</sup>	nd	nd	nd
54	1419	3-ethyl-2-methyl-1,3-hexadiene	67	3	16.4 $\pm$ 0.1 <sup>a</sup>	nd	19.2 $\pm$ 0.1 <sup>b</sup>	nd	nd	nd
55	1473	nerol oxide	68	2	0.43 $\pm$ 0.10	nd	0.50 $\pm$ 0.07	nd	5.35 $\pm$ 0.01 <sup>a</sup>	5.68 $\pm$ 0.01 <sup>b</sup>
56	1548	linalool	71	1	28.2 $\pm$ 0.5	29.4 $\pm$ 0.7	29.2 $\pm$ 0.7	10.2 $\pm$ 0.4 <sup>b</sup>	8.12 $\pm$ 0.31 <sup>a</sup>	7.78 $\pm$ 0.05 <sup>a</sup>
57	1610	hotrienol	71	1	28.7 $\pm$ 1.0 <sup>a</sup>	39.5 $\pm$ 1.7 <sup>b</sup>	nd	tr	tr	nd
58	1698	$\alpha$ -terpineol	59	1	0.54 $\pm$ 0.06 <sup>a</sup>	0.47 $\pm$ 0.03 <sup>a</sup>	0.85 $\pm$ 0.02 <sup>b</sup>	1.55 $\pm$ 0.03 <sup>b</sup>	0.24 $\pm$ 0.02 <sup>a</sup>	1.50 $\pm$ 0.04 <sup>b</sup>
59	1724	lilac alcohol	55	2	nd	0.14 $\pm$ 0.09	nd	nd	nd	nd
60	1735	neral	69	1	nd	31.2 $\pm$ 0.9 <sup>b</sup>	26.2 $\pm$ 1.1 <sup>a</sup>	46.1 $\pm$ 0.1 <sup>a</sup>	51.5 $\pm$ 0.3 <sup>b</sup>	51.8 $\pm$ 0.0 <sup>b</sup>
61	1764	$\beta$ -citronellol	69	1	tr	tr	tr	0.91 $\pm$ 0.13 <sup>a</sup>	1.07 $\pm$ 0.03 <sup>a</sup>	2.25 $\pm$ 0.08 <sup>b</sup>
62	1799	nerol	69	2	157 $\pm$ 1 <sup>a</sup>	169 $\pm$ 1 <sup>c</sup>	164 $\pm$ 0.4 <sup>b</sup>	65.5 $\pm$ 0.4 <sup>a</sup>	101 $\pm$ 4 <sup>b</sup>	170 $\pm$ 1 <sup>c</sup>
<b>Pyrazine</b>										
63	1385	2-ethyl-6-methylpyrazine	121	1	25.1 $\pm$ 1.0 <sup>a</sup>	86.7 $\pm$ 18.2 <sup>b</sup>	72.0 $\pm$ 5.5 <sup>b</sup>	nd	nd	nd
64	1435	2,6-diethylpyrazine	135	1	24.7 $\pm$ 2.0 <sup>a</sup>	41.0 $\pm$ 5.6 <sup>b</sup>	19.2 $\pm$ 0.2 <sup>a</sup>	nd	nd	nd
65	1462	3-ethyl-2,5-dimethylpyrazine	135	1	22.0 $\pm$ 0.2 <sup>a</sup>	106 $\pm$ 4 <sup>c</sup>	68.2 $\pm$ 5.5 <sup>b</sup>	nd	nd	nd
<b>Furan</b>										
66	1224	2-pentylfuran	81	2	9.99 $\pm$ 0.16 <sup>a</sup>	16.1 $\pm$ 0.5 <sup>b</sup>	18.6 $\pm$ 0.2 <sup>c</sup>	nd	nd	nd
67	1469	furfural	96	1	82.9 $\pm$ 9.8 <sup>a</sup>	188 $\pm$ 21 <sup>b</sup>	117 $\pm$ 1 <sup>a</sup>	nd	nd	nd
68	1509	1-(2-furanyl)-ethanone	95	2	9.75 $\pm$ 1.06 <sup>a</sup>	12.9 $\pm$ 0.4 <sup>b</sup>	11.5 $\pm$ 0.6 <sup>ab</sup>	nd	nd	nd
69	1578	5-methyl-2-furfural	110	1	10.5 $\pm$ 0.4 <sup>a</sup>	21.2 $\pm$ 1.9 <sup>b</sup>	14.3 $\pm$ 0.0 <sup>a</sup>	nd	nd	nd
<b>Phenol</b>										
70	2010	phenol	94	1	5.17 $\pm$ 0.83 <sup>ab</sup>	7.13 $\pm$ 1.04 <sup>b</sup>	2.65 $\pm$ 0.31 <sup>a</sup>	69.5 $\pm$ 2.1 <sup>b</sup>	44.0 $\pm$ 9.4 <sup>a</sup>	65.2 $\pm$ 2.2 <sup>ab</sup>
71	2146	4-ethenyl-2-methoxyphenol	135	2	1.28 $\pm$ 0.00 <sup>a</sup>	4.40 $\pm$ 0.10 <sup>b</sup>	4.13 $\pm$ 0.26 <sup>b</sup>	5.81 $\pm$ 0.39 <sup>a</sup>	27.9 $\pm$ 8.1 <sup>b</sup>	4.04 $\pm$ 0.13 <sup>a</sup>

TSR: Thompson seedless raisins; FSR: Flame seedless raisins; CSR: Crimson seedless raisins. <sup>A</sup> Mean  $\pm$  standard deviation ( $n = 3$ ) of the same compounds followed by different lowercase superscripts are significantly different ( $p < 0.05$ ), the free and bound volatiles were compared separately. tr: LOD < concentration < LOQ. nd: concentration < LOD. <sup>B</sup> The characteristic ion ( $m/z$ ) was used for choosing the corresponding compound in order to avoid possible interference by other compounds. <sup>C</sup> Identification method: 1, identified, mass spectrum and RI were in accordance with standards; 2, tentatively identified, mass spectrum matched in the standard NIST 2008 Library and RI matched with the NIST Standard Reference Database (NIST Chemistry Web Book); 3, tentatively identified, mass spectrum agreed with the standard NIST 2008.



**Figure 1.** Schematic representation of cast-tape drying (CTD) experimental device (adapted from Durigon *et al.*, 2018).



**Figure 2.** Schematic representation of refractance window drying system for past and purées (adapted from Caparino *et al.*, 2012).



### Spray-drying

The spray-drying of soursop (*Annona muricata* L.) on a laboratory dryer was reported by Neta *et al.* (2019), which generated 85 volatile compounds. Of these compounds, the aliphatic esters accounted for approximately 78% or 173 mg kg<sup>-1</sup> of the identified compounds, followed by terpenes, acids, and alcohols. Spray-drying noticeably reduced the volatile compounds of the fresh fruit. For example, some compounds such as *cis*-11-hexadecenal, (*Z,E*)- $\alpha$ -bergamotol, ethyl-2-hexanoate, phenyl ethanal, (*Z*)-7-hexadecenal, (*Z*)-9-hexadecenal, acetic acid, pentadecanoic acid, methyl-4-oxohexanoate, ethyl octanoate, dimethyl hexanedioate, methyl vanillate,  $\alpha$ -pinene,  $\alpha$ -ocimene, and farnesane which were identified in the fresh fruit were not found in the dried powdered fruit (Neta *et al.*, 2019). Overall, quite significant amount of volatiles were lost (73%) in the rehydrated soursop powder as a result of spray-drying. The probable reasons for the large losses of volatile compounds might be due to the retention of particulates within the spray-dryer (Forero *et al.*, 2015; Durigon *et al.*, 2018).

### Freeze-drying

#### Red guava (*Psidium guajava* L. cv. Pedro Sato)

The dehydration of fresh guava using a freeze-dryer was reported by Nunes *et al.* (2016), which generated 31 volatile compounds. The volatile compounds were made up of 16 terpenes, six esters, and five aldehydes, respectively. Alpha-humulene and  $\beta$ -caryophyllene were the major volatile compounds in both fresh and freeze-dried guava samples, and they contributed altogether approximately 34 - 48% of the total volatiles' content. Beta-caryophyllene which is usually found in combination with its isomer,  $\alpha$ -humulene, is a key aroma compound of guava fruit (Pino and Bent, 2013). Freeze-drying of guava did not alter the characteristic fresh guava aroma. This is reflected in the high relative contents of compounds such as (*Z*)-3-hexenyl acetate, hexanal, and (*E*)-2-hexenal. However, freeze-drying caused appreciable reduction in the concentrations of some compounds such as  $\alpha$ -pinene oxide, ethyl acetate, caryophyllene oxide, (*Z*)-3-hexenyl butyrate, (*Z*)-3-hexenyl acetate, hexyl acetate, (*Z*)-3-hexenyl hexanoate, hexanal, (*E*)-2-hexenal, and 2-nonenal. Most probable reasons for this observation might be due to the high vapour pressure within the freeze-dryer (Nunes *et al.*, 2016).

### Pear (*Pyrus communis* L.)

The dehydration of pear pureés in a freeze-dryer was reported by Komes *et al.* (2007), which generated 19 volatile compounds. Of these, methyl butanoate, propyl acetate, ethyl butanoate, hexanal, butyl acetate, 2-hexenal, *n*-amyl acetate, and hexyl acetate accounted for approximately 80% of the compounds. It is noteworthy that the retention of volatile compounds in freeze-dried pureés is directly related to the dehydration method and the type of sugars added prior to dehydration.

### Withering

The dehydration of Erbaluce grape cultivar was reported by Rolle *et al.* (2012). The natural off-vine withering process of Erbaluce grapes resulted in noticeable changes in the colour of some berry clusters. Some berries changed from their initial green-yellow colour to green, gold, and blue grapes, respectively. These changes are caused by the disintegration of those proteins which already exist within the cell of the grape and moulds (Rolle *et al.*, 2012). In that study, a total of 58 free volatile compounds belonging to 11 different classes were reported in the three types of dehydrated grapes. The blue dehydrated grapes volatiles were dominated majorly by acids, terpenoids, norisoprenoids, ethyl, and methyl esters as compared to volatiles of the other grapes. The dehydration of the grapes engendered the formation of norisoprenoids which were formed via the oxidation of carotenoids (Rolle *et al.*, 2012). The dehydration of infected berries by *Botrytis cinerea* resulted in these blue grapes as well as the formation of C-18 unsaturated long-chain esters,  $\delta$ -lactones, 3-furanacetic acid, and methyl benzeneacetate. Other compounds identified in the blue grape were *cis*-bisabolene, indipone, phenol, and glycerol. In addition,  $\delta$ -lactones were the most abundant volatile compounds, accounting for 67%, followed by esters (32%). Meanwhile, the alcohols accounted for 54% (w/v) of the total volatiles in the green grapes. Other major volatile compounds in the green Erbaluce dehydrated grapes were the methyl hexadecanoate, ethyl hexadecanoate, and benzenoids. These were followed by homovanillic acids, terpenoids, and ketones. In the case of the third type of grape (*i.e.*, gold dehydrated grapes), there was a significant increase in the level of hexanoic acid, and a slight reduction in the levels of total esters as compared to the other types of grapes. Overall, the gold

dehydrated grapes produced similar volatile profile to that of the green grapes.

A summary of the results of research

conducted using other drying technologies on the volatile compounds' retention or losses in dehydrated fruits are further presented in Table 2.

**Table 2.** Summary of the results of research conducted using other drying technologies on the volatile compounds' retention or losses in dehydrated fruits.

Fruit	Drying technology	Critical findings on volatile compound	Reference
Pear	Foam-mat	<ul style="list-style-type: none"> <li>In foam-mat dried pear puree without sugar addition, all esters, except butyl butanoate, were retained in the average percentage of 33%.</li> <li>Foam-mat drying was only able to retain 31% of the fresh fruit aroma compounds.</li> </ul>	Komes <i>et al.</i> (2007)
Balsam pear	Microwave	<ul style="list-style-type: none"> <li>The following compounds were newly generated and varied with drying time, for example, ethyl lactate, 2-pentene-1-ol, ethylbenzene, and xylene.</li> <li>Some compounds appeared when the samples were burned, such as cis-decahydro-1-methyl-quinoline.</li> </ul>	Li <i>et al.</i> (2021)
Figs	Sun drying	<ul style="list-style-type: none"> <li>Ethyl alcohol, isopentyl alcohol, isopentyl alcohol acetate, ethyl acetate, 3-methylbutanal, hexanal, and benzaldehyde were the major compounds in both fresh and dried Dottato figs.</li> </ul>	Russo <i>et al.</i> (2017)
Sapodilla	Oven drying	<ul style="list-style-type: none"> <li>Twenty-nine aroma-active compounds were quantified in fresh and dried sapodilla fruits.</li> <li>Fresh fruit showed higher potency for ethyl benzoate, E-2-hexenal, and <math>\beta</math>-caryophyllene.</li> <li>The dried fruit exhibited very high potency for <math>\alpha</math>-sinensal, and to a lesser extent, E-2-hexenal, ethyl benzoate, <math>\beta</math>-caryophyllene, and hexyl benzoate.</li> </ul>	Lasekan and Yap (2018)
Tomato	Freeze drying	<ul style="list-style-type: none"> <li>An increase in contents of compounds such as dimethyl sulphide, 2-ethyl furan, hydroxymethyl furfural, acetaldehyde, and <math>\alpha</math>-terpineol was found in all dehydrated products.</li> <li>Compounds such as 3-methyl furan, hexanol, and terpinyl acetate were found to occur only in dehydrated products.</li> <li>The presence of <math>\gamma</math>-undecalactone (fruity odour note) was identified for the first time in fresh and all dehydrated tomatoes.</li> </ul>	Rajkumar <i>et al.</i> (2021)
Moscato bianco' grapes	Cane-cut on-vine	<ul style="list-style-type: none"> <li>Increased glycosidically-bound volatile compounds than in the free fraction was reported.</li> <li>Bound linalool showed a significant increase of 52% when cane-cut withering system was applied.</li> <li>There was a significant glycosylated forms of nerol and geraniol observed in the two on-vine withering systems at 24<sup>th</sup> day vs control.</li> </ul>	Giacosa <i>et al.</i> (2019)
Strawberries	Osmotic dehydration	<ul style="list-style-type: none"> <li>Osmotic treatments provoked a loss in volatile compounds due to the migration, mainly of esters, into the osmotic media.</li> <li>The variations in the volatile pattern depended on both time of treatment and type of osmotic solution.</li> <li>The greater changes occurred after 2 h in sucrose; with a promotion of fermentative volatiles (acetaldehyde, ethyl acetate), and a decrease in the other volatiles.</li> </ul>	Rizzolo <i>et al.</i> (2007)
Pineapple	Conductive multflash drying	<ul style="list-style-type: none"> <li>Drying processes caused changes in the volatile compounds' profile. Part of the compounds detected in the pineapple pulp samples were lost during the drying.</li> <li>On the other hand, the hexadecane content showed a significant increase after drying, corresponding to 57.42 and 85.52% of the total volatile fraction in the pineapple product.</li> <li>Some compounds were only identified in the dried samples, such as 2,5-dimethyl-4-hydroxy-3(2H)-furanone (furanol) and acetic acid.</li> </ul>	Simao <i>et al.</i> (2021)

Tomato	Thin-layer catalytic far-infrared radiation drying	<ul style="list-style-type: none"> <li>• Application of FIR enhanced the flavour of the dried tomatoes by 36.6% when compared with the raw ones.</li> <li>• Enhancement in flavour.</li> <li>• Improvements in the quality and functional property of dried tomatoes were produced.</li> </ul>	Abano <i>et al.</i> (2014)
Mango	A pilot unit (UTA dryer, Villeneuve-sur-Lot, France)	<ul style="list-style-type: none"> <li>• Drying resulted in substantial losses of most compounds.</li> <li>• The total amount of volatiles decreased by about 59%.</li> <li>• New compounds appeared and enrichment of some compounds was observed after drying.</li> <li>• Limonene, <math>\beta</math>-myrcene, <math>\delta</math>-3-carene, <math>\beta</math>-caryophyllene, <math>\gamma</math>-butyrolactone, and 3-methylbutyl butanoate were found to be flavour contributors in both fresh and dried mangoes.</li> </ul>	Bonneau <i>et al.</i> (2016)

### Challenges and future perspective

In recent years, drying has grown to be a prime operation in the food industry with several drying methods being used. Of these is the conventional hot air-drying. This method exposes fruit to high temperatures which often results in high losses of volatile constituents and the formation of off-flavour compounds. Moreover, the best drying method, which is the freeze-drying, is fraught with problems of high maintenance, end-product cost, and the production of significant losses of bioactive compounds. However, a novel drying technology, Refractance Window™ drying has gained huge attention due to its several advantages which includes the ability to dry heat-sensitive products, retention of fruit's colour and aroma, it dries the product through a thin, transparent infrared film which forms a 'window' for drying, and the drying cost and energy consumption are lower than conventional drying technologies. The ability of this technology to produce high quality dried fruits with high aroma compound retention is a pointer to its potential scale up for industrial fruit processing.

### Conclusion

Dehydration processes result in noticeable losses/reduction of volatile compounds such as monoterpenes, sesquiterpenes, aliphatic alcohols, esters, and lactones, which are considered as impact odorants. Dehydration processes also result in the production of some compounds either via (1) hydrolysis of relevant glycosides under high temperatures, or (2) thermal degradation of volatile and non-volatile precursors, as well as oxidation and Maillard reactions, which result in the production of heterocyclics, and saturated and unsaturated aldehydes. Another dehydration process namely grape withering significantly improves the development of fruit aroma, and contributes to

carotenoid oxidation, thus leading to the formation of the pleasant-smelling norisprenoids. Of significance is the Refractance Window™ drying which exhibits high retention potential (~ 90%) of volatile compounds present in fresh fruits. This drying technology produces rapid food drying at very low temperature.

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