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Effect of combined drying on the physical properties and bioactive components of 'Jonathan' apple (*Malus domestica* L.)

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ABSTRACT

The main objectives of the present study were to investigate the physical attributes and chemical characteristics of dried apple cubes. The apple cubes were dried in vacuum and infrared dryers at 40 °C and freeze dryer individually and in combination. The physical properties – texture and rehydration – of vacuum drying-assisted freeze-dried apple dices were close to the freeze-dried products. The hardness of vacuum drying-assisted freeze-dried apples was better, by at least 15.8%, than those dried by infrared-freeze, but rehydration capacities were similar. The freeze-dried control samples achieved a lower water activity (0.145) in comparison with infrared, vacuum, and combined dried ones. The infrared-freeze-dried samples retained 3.6 and 11.6% more polyphenols and antioxidant capacity than samples prepared by the other methods. The ascorbic acid content of the freeze-dried samples was significantly higher by about 59% than that of the hybrid dried samples.

KEYWORDS

hybrid drying, water activity, rehydration, texture, phenolics, antioxidant activity

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1. INTRODUCTION

Raw apple is a perishable food due to its high water content (80–88% w.b.), so it needs to be preserved. The drying methods influence both the physical and chemical characteristics of the dried products.

Freeze-dried products are scientifically proven to be of high quality. Freeze drying (FD) requires a long drying time to remove water from fruit and vegetables; it is a relatively slow drying process (Shewale et al., 2019). Dehydration is a very energy-intensive process, so there is a strong focus on minimising the energy demand of drying. One possible solution for reducing the operating time of lyophilisation is to combine it with drying methods with fast water removal ability while being gentle for the quality of the finished product (Antal, 2015). Infrared drying (IRD) is a low-cost drying technique. In IRD, the heating element transfers radiant energy to the surface of the food and the energy penetrates the product without heating the surrounding air (Hebbar and Rastogi, 2001). The infrared energy is rapidly absorbed by the water molecules in the food product, resulting in rapid evaporation of the water (Sonawane and Arya, 2015). The combination of IRD with other dehydration methods is a promising process and has been shown to be a potential drying method for many food products, as it not only increases the drying rate but also the quality of the food (Jeevarathinam et al., 2021; Delfiya et al., 2022). For the vacuum drying technique (VD), the relation between the product to be dehydrated and the oxygen is limited. The reduced pressure ensures efficient drying at low temperatures (Nawirska et al., 2009).

The literature contains references to the drying of apple cubes by the infrared-assisted freeze-drying method, but no reports are available on the drying of apples dehydrated by combined vacuum drying-freeze drying and the effect of drying on the main characteristics of apples. The major objective of this work was to study the effect of various hybrid drying methods like vacuum drying-assisted freeze drying and infrared drying-assisted freeze drying on the physical and chemical characteristics of apple dices. The quality of dried apple cubes was determined in terms of rehydration capacity, texture, moisture content, water activity, phenolic content, antioxidant activity, and ascorbic acid content. In addition, the aim of this scientific work was to investigate whether traditional gentle freeze drying can be replaced by hybrid drying methods.

2. MATERIALS AND METHODS

2.1. Sample preparation

The samples (*Malus domestica* cv. Jonathan) were washed with tap water, peeled, cored with a knife, and then cut into cubes of 10 mm thickness using a hand operated slicer.

The mass of sample was measured on an analytical balance (model JKH-500, Jadever, New Taipei, Taiwan) with a precision of ± 0.1 g.

Before drying, the samples were pretreated to avoid enzymatic browning. The concentration of the citric acid solution used was 1%.

2.2. Determination of moisture content

The initial and final moisture contents of apple cubes were determined using the gravimetric method at 105 °C for 25 h in a dryer (LP-302, Kapacitív Ltd., Budapest, Hungary). The initial moisture content of samples was 6.092 ± 0.123 kg H_2O/kg d.m. (85.9 ± 1.1% w.b.).

2.3. Dehydration procedure

For each experiment, 100 ± 0.5 g apple samples were used placed in a single layer on the tray.

Infrared drying (IRD): A lab scale infrared dryer (model Precisa HA-60, Precisa Gravimetrics AG, Dietikon, Switzerland) equipped with 2 quartz glass infrared emitter (a total of 410 W) was used. The apples were dried using a wavelength of 1.4–3 μm, so called MIR (middle-infrared) radiation.

The drying air temperature was measured using a temperature probe (model Testo 4510, Testo GmbH, Germany). The tray was connected to the electronic balance with precision of ± 0.01 g (model Precisa, Precisa Gravimetrics AG, Dietikon, Switzerland).

Vacuum drying (VD): The apple samples were dried in a vacuum (model Kambic VS-50C, Kambic Lab. Eq., Semic, Slovenia) at 40 °C. The pressure in the vacuum dryer was 5 kPa during the drying process.

The mass of the sample was measured during drying by a digital balance (model JKH-500) of 500 g weighing capacity and with an accuracy of ±0.1 g. The electronic balance was placed outside the drying chamber.

Freeze drying (FD): Apple cubes were dried for 22 h using an Armfield FT-33 laboratory freeze dryer (Armfield Ltd., Ringwood, UK). The freeze-drying process is described in a previous publication (Antal, 2015).

Hybrid drying (IRD-FD and VD-FD): The drying parameters set for the combined methods were the same as those described for IRD, VD, and FD dryers.

IRD-FD: The combined dehydration experiments started in an infrared apparatus (pre-drying) with drying times of 4 and 6 min, the moisture content of the material reached 57 ± 0.5% and 46 ± 0.5% (w.b.). The drying process of the apple cubes continued in the freeze dryer (post-drying) until constant weight was reached.

VD-FD: The apple cubes were placed in a vacuum dryer and pre-dried until the water content of the dehydrated samples reached 55 ± 0.5% and 42 ± 0.5% (w.b.), this corresponds to drying times of 2 and 3 h. Then, the apple dices were further dried using a freeze dryer.

The main parameters of the drying experiments are given in Table 1. The drying process was continued until no further changes in the weight of the apple samples were observed.

2.4. Rehydration experiments

Rehydration of dried apple was performed in liquid medium at 30 °C for 30 min. Approximately 0.5 g (± 0.01 g) of dried sample was placed in a 100 mL laboratory plastic vessel, and 80 mL distilled water was added. The rehydrated sample was then removed from the vessel, drained for 10 s on a filter paper, and weighed on a digital balance with precision of ±0.01 g (model Precisa). The rehydration ratio (RR) was calculated according to Eq. (1):

$$RR = \frac{W_{reh}}{W_{dry}}, \tag{1}$$

where: W_{reh} is weight of rehydrated sample (g), W_{dry} is weight of dried sample (g).

Table 1. The drying parameters of apple cubes

Drying methods	Drying temperature (°C)	Pre-drying time (min)	Pre-drying temperature (°C)
FD	−25–20	–	–
IRD	40	–	–
VD	40	–	–
4min-IRD-FD	–	4	40
6min-IRD-FD	–	6	40
2h-VD-FD	–	120	40
3h-VD-FD	–	180	40

FD: freeze drying; IRD: infrared drying; VD: vacuum drying; IRD-FD: infrared-freeze drying; VD-FD: vacuum pre- and freeze finish drying.

2.5. Hardness test

Measurements were performed using a texture analyser (model CT3-4500, Brookfield Engineering Laboratories, Middleboro, USA). The maximum compression force of material was used to describe the sample texture in terms of hardness or firmness (reported in Newton). The following setting parameters were used: 4.5 kg force load cell, 2 mm s^{-1} test speed, 20 mm travel distance, and 4 mm diameter of cylindrical probe. The maximum depth of penetration was 3 mm and the trigger force was 10 g.

2.6. Water activity assay

The water activity (a_w) of fresh and dried apple was evaluated at 23 °C using a water activity meter (model Novasina Labmaster CH-8853, Novasina AG, Switzerland). Approximately 2 g of the chopped apple sample was put in the chamber of the equipment.

2.7. Sample preparation for determination of the bioactive components

The dried samples were milled using a laboratory grinder (model QC-124, Kapacitív Ltd, Budapest, Hungary). The extraction of polyphenol compounds was carried out using the method proposed by Chong et al. (2014).

2.8. Determination of total phenolic content

The total phenolic content (TPC) was measured using the Folin–Ciocalteu method of Vega-Gálvez et al. (2012). The absorbance was measured at 765 nm using a UV–Vis spectrophotometer (Perkin Elmer Lambda 35, PerkinElmer, USA) after 60 min using gallic acid as standard. The results were expressed as gallic acid equivalents (GAE), mg g^{-1} of dry matter.

2.9. Determination of antioxidant activity

The DPPH free radical scavenging activity of the samples was determined using the method according to the work of Chong et al. (2014). The absorbance was measured at 517 nm using a UV–Vis spectrophotometer (Perkin Elmer Lambda 35, PerkinElmer, USA). The results were corrected for dilution and expressed in μM Trolox equivalents (TE)/g of dry mass.

2.10. Determination of ascorbic acid content

Ascorbic acid content of apple was determined by the dichlorophenol-indophenol titrimetric method. Ascorbic acid content was determined following a procedure previously described by the authors (Shewale and Hebbar, 2017). Ascorbic acid content was expressed as mg of ascorbic acid equivalents/100 g of dried mass.

2.11. Statistical analysis

Each experiment was repeated three times and the average value is shown. All experiments were subjected to analysis of variance (ANOVA) test using IBM SPSS Statistics version 20.0 (IBM Inc., Armonk, NY, USA). Means were compared by using Duncan's multiple range test at a confidence level of 95%, where $P < 0.05$ represents a significant difference. Results in tables and graphs are presented as the mean ± standard deviation (SD) of three independent determinations.

3. RESULTS AND DISCUSSION

3.1. Influence of different drying methods on drying time and water activity

Figure 1 shows the effect of single and two stage drying methods on the drying time. The required drying times to reach mean final moisture contents of 0.099–0.141 kg H_2O/kg (d.b.) for FD, IRD, VD, 4min-IRD-FD, 6min-IRD-FD, 2h-VD-FD, and 3h-VD-FD were 1320, 34, 570, 784, 666, 900, and 840 min, respectively.

The operating time of the IRD method is 2.57% of the drying time of the FD. Similarly, the VD dehydration method is 43.18% of the operating time of the FD. As can be observed in Fig. 1, the combined drying procedure (IRD-FD and VD-FD) decreased significantly ($P < 0.05$) the freeze drying (FD) time by up to 49%, due to the increase of drying rate in the first period of drying. In the present study, the drying times measured for infrared and vacuum assisted drying were around 31.8–49.5% lower than the FD drying time. Shih et al. (2008) tested infrared-assisted drying, which resulted in a reduction in the drying time of 42% as compared to freeze drying. As expected, the operating time decreased during combined drying when the drying time increased from 4 to 6 min and from 2 to 3 h. No significant difference ($P > 0.05$) was found between the operating times of the 2h-VD-FD and 3h-VD-FD drying methods. The results showed that infrared pre-drying can promote apple cube dehydration better ($P < 0.05$) than the VD-FD method during combined drying.

The moisture content and water activity values for each product are shown in Table 2. The moisture content and water activity (a_w) of the fresh apple cubes were 85.9% (w.b.), 6.092 kg H_2O/kg (d.b.), and 0.969, respectively. Food stability and safety are greatly improved if the a_w of the dried product is reduced below 0.6 (Fontana, 2000). The lowest moisture content and a_w values were observed for freeze-dried apple (0.099 kg H_2O/kg and 0.145), which ensures high stability during storage. Similar results were observed in the study of Kahraman et al. (2021). The data revealed no significant difference ($P > 0.05$) observed in the water activity of IRD, VD, 6-min-IRD-FD, and 2h-VD-FD dried samples. The a_w value of apple cubes preserved by all drying methods was below 0.23, i.e. microbiologically stable.

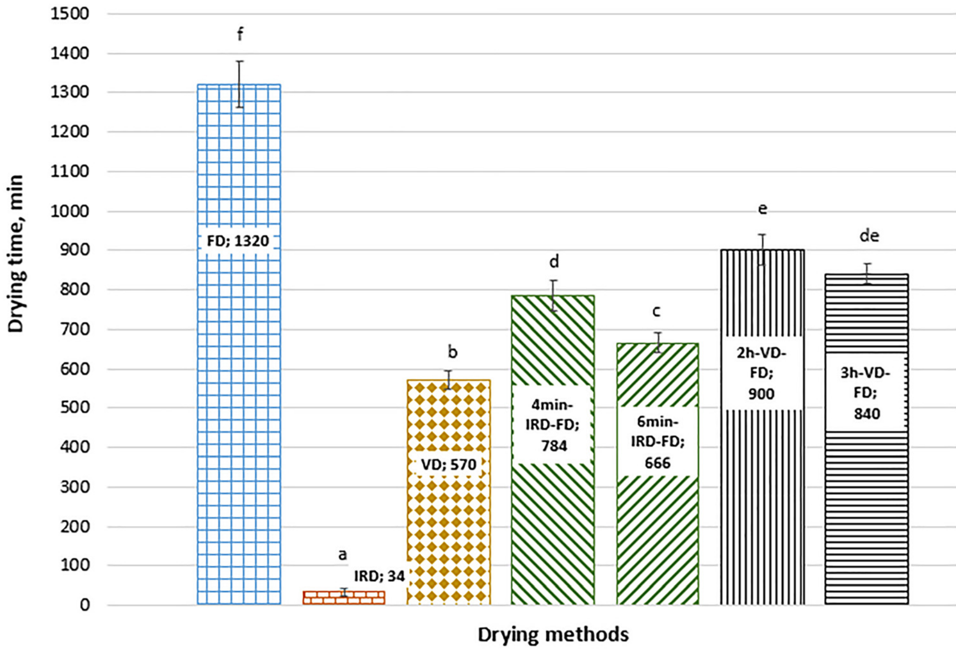

Fig. 1. Operational times of one- and two-stage dehydration methods for drying apples

	FD	IRD	VD	4minIRD-FD	6minIRD-FD	2hVD-FD	3hVD-FD
Initial moisture content (kg H_2O/kg d.m.)	6.092	6.092	6.092	6.092	6.092	6.092	6.092
Final moisture content (kg H_2O/kg d.m.)	0.099	0.107	0.115	0.141	0.120	0.106	0.129
Drying temperature (°C)	–25–20	40	40	40; 20	40; 20	40; 20	40; 20

FD: freeze drying; IRD: infrared drying; VD: vacuum drying; IRD-FD: infrared-freeze drying; VD-FD: vacuum pre- and freeze finish drying
Data are expressed as the average ± standard deviation for three replicates.
Different lowercase letters (a, b, c, d, e, f) denote significant differences ($P \leq 0.05$)

The moisture content of apple samples treated with one- and two-stage drying was 1.4–2% (w.b.) expressed on a wet basis (Table 2). It is observed that there is a linear relationship between the water activity values and the moisture content of the finished product.

Table 2. Effect of different drying methods on water activity and moisture content of apple cubes

Drying methods	Water activity, a_w (–)	Moisture content, M (kg H_2O/kg d.m.)
Fresh	0.969 ± 0.11^{e}	6.092 ± 0.123^{f}
FD	0.145 ± 0.04^{a}	0.099 ± 0.004^{a}
IRD	0.158 ± 0.03^{b}	0.107 ± 0.006^{b}
VD	0.163 ± 0.05^{b}	0.115 ± 0.005^{c}
4min-IRD-FD	0.221 ± 0.06^{d}	0.141 ± 0.009^{e}
6min-IRD-FD	0.168 ± 0.03^{b}	0.120 ± 0.005^{c}
2h-VD-FD	0.159 ± 0.05^{b}	0.106 ± 0.008^{b}
3h-VD-FD	0.187 ± 0.04^{c}	0.129 ± 0.007^{d}

FD: freeze drying; IRD: infrared drying; VD: vacuum drying; IRD-FD: infrared-freeze drying; VD-FD: vacuum pre- and freeze finish drying.
Data are expressed as the average ± standard deviation for three replicates. Values in the same column with different superscripts (a, b, c, d, e, f) are significantly different ($P \leq 0.05$).

3.2. Rehydration characteristics of dried apple cubes

Rehydration ratio (RR) values of the dried apple samples by different drying methods are shown in Fig. 2. The rehydration ratio for FD, IRD, VD, 4min-IRD-FD, 6min-IRD-FD, 2h-VD-FD, and 3h-VD-FD were 6.35, 3.67, 4.24, 5.84, 5.83, 6.05, and 5.99, respectively.

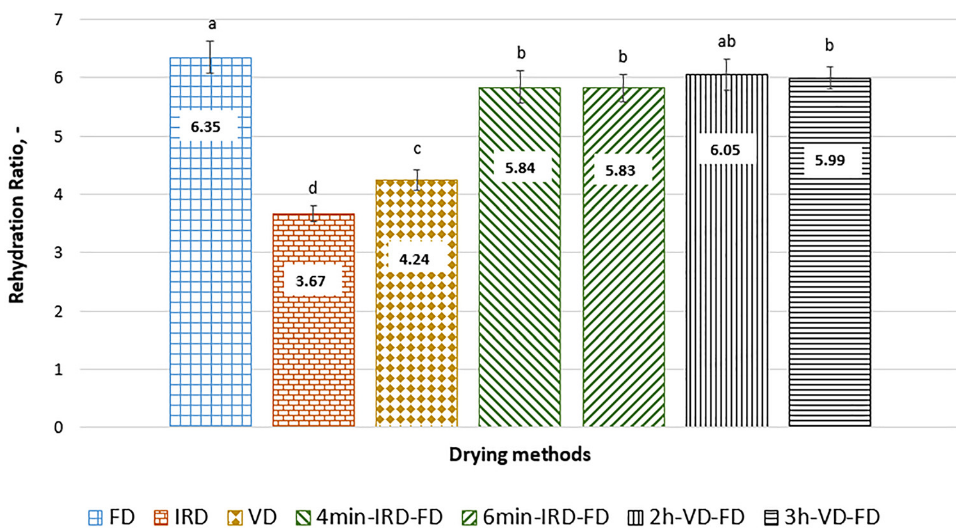

Fig. 2. Effect of different drying methods on rehydration of apple cubes
FD: freeze drying; IRD: infrared drying; VD: vacuum drying; IRD-FD: infrared-freeze drying; VD-FD: vacuum pre- and freeze finish drying
Data are expressed as the average ± standard deviation for three replicates.
Different lowercase letters (a,b,c,d) denote significant differences ($P \leq 0.05$)

The apple samples treated with conventional vacuum and infrared drying had the lowest RR values, and there was a significant difference ($P < 0.05$) between them. The highest rehydration rate values were observed for the FD and 2h-VD-FD apple cubes, no significant difference ($P > 0.05$) was found between them. The higher RR in the FD and 2h-VD-FD samples can be explained by the formation of large pores, which are able to absorb large amounts of wetting medium (Tüfekçi and Özkal, 2017).

Increasing the drying time during pre-drying (from 4 to 6 min at IRD and from 2 to 3 h at VD) did not result in a change in rehydration capacity. No significant difference ($P > 0.05$) was observed between the rehydration rate values of apple cubes dried by the combined methods, although the RR value of VD-FD samples was higher than that of IRD-FD samples.

3.3. Texture strength of dried apple cubes

The effect of single-stage and two-stage drying methods on the hardness of the dried product is shown in Fig. 3. In the present study, the hardness values of apple dices dried by FD, IRD, VD, 4min-IRD-FD, 6min-IRD-FD, 2h-VD-FD, and 3h-VD-FD were 4.51 N, 9.15 N, 7.87 N, 6.28 N, 5.63 N, 4.82 N, and 4.74 N, respectively.

The hardness parameter of the lyophilised (FD) and vacuum pre- and finish-freeze dried (VD-FD) samples was significantly lower ($P < 0.05$) than that of the raw material. Statistically, there was no significant difference ($P > 0.05$) among the hardness of FD, 2h-VD-FD, and 3h-VD-FD apple cubes, but the hardness of FD apple samples was lower. The FD apple cubes showed the lowest firmness of all samples, which indicates that the FD products had a soft

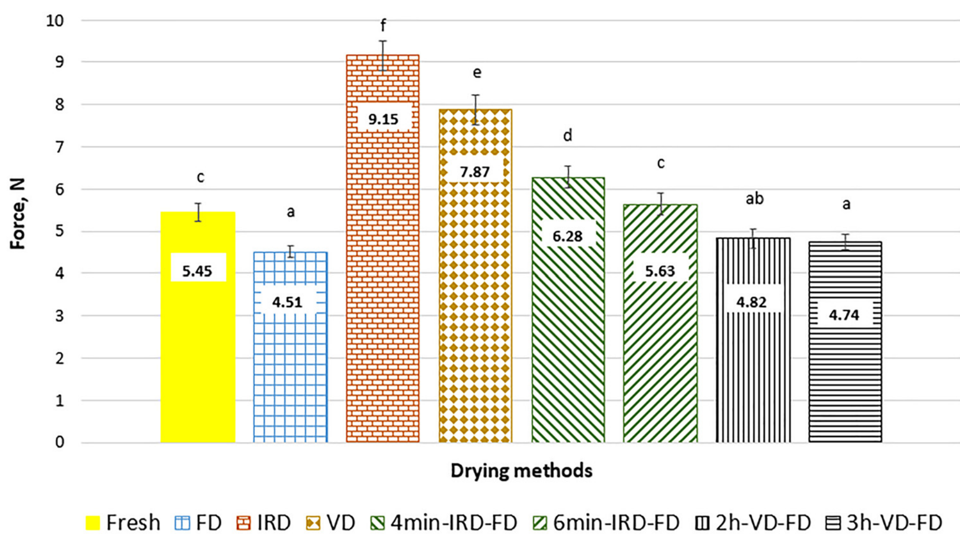

Fig. 3. Texture of fresh and apple dices dried by different methods
FD: freeze drying; IRD: infrared drying; VD: vacuum drying; IRD-FD: infrared-freeze drying; VD-FD: vacuum pre- and freeze finish drying
Data are expressed as the average ± standard deviation for three replicates.
Different lowercase letters (a, b, c, d, e, f) denote significant differences ($P \leq 0.05$)

texture. The FD caused little injuries in the apple, resulting a thin cell wall. This thin cell wall was poorly resistant to external forces, which reduced the firmness of the lyophilised apple (Cui et al., 2018). It was found that the firmness values of the 6min-IRD-FD samples were close to those of the fresh samples (5.45 N), with no significant difference ($P > 0.05$) between them. An increase in pre-drying time (from 4 to 6 min and from 2 to 3 h) under IRD-FD and VD-FD decreased the hardness of the product, although there was no significant difference ($P > 0.05$) in hardness between 2h-VD-FD and 3h-VD-FD apple cubes. It also shows that the hardness of the apple samples under VD-FD was significantly softer ($P < 0.05$) than IRD-FD ones.

The traditional infrared (IRD) and vacuum drying (VD) methods had a negative effect on the structure and texture of the apples, as they had the highest resistance to the penetrometer.

3.4. Effect of drying on bioactive components of dried apple dices

The influence of single-stage and two-stage drying on the content of phenolic compounds, antioxidant capacity, and ascorbic acid content of apple is presented in Table 3.

The 6min-IRD-FD, FD, 2h-VD-FD, 4min-IRD-FD, 3h-VD-FD, IRD, and VD dried apple dices showed 11.5, 10.3, 8.5, 7.3, 6.0, 5.6, and 5.4 μM Trolox/g antioxidant activity on dry weight basis, respectively. The antioxidant activity (AA) retention of 6min-IRD-FD apple cubes was significantly higher ($P < 0.05$) than that obtained for FD ones. In the present study, increase in pre-drying time (from 4 to 6 min) of IRD-FD increased the antioxidant activity (AA), total phenolic content (TPC), and vitamin C content in the samples. In the case of combined drying methods, the pre-drying time had a significant effect on the bioactive components of the food. This fact is described in a previous study on the effect of different settings of hot air pre-drying and vacuum-microwave finish-drying on the antioxidant capacity and phenolic compounds of garlic (Calín-Sánchez et al., 2014). The VD-FD combined drying method showed that an increase in pre-drying time (from 2 to 3 h) had the opposite effect on the TPC, AA content, and vitamin C content of apples when compared to IRD-FD. In fact, no significant ($P > 0.05$) differences were found between the TPC, and AA of IRD and VD apple samples. As can be seen in Table 3, the total phenolic content directly correlated with the antioxidant activity.

Table 3. Effect of different drying techniques on antioxidant activity, total phenolic content, and ascorbic acid content of dehydrated apple dices

Drying methods	Antioxidant activity (AA) (μM TE g^{-1})	Total phenolic (TPC) (mg GAE g^{-1})	Ascorbic acid (mg 100 g^{-1})
FD	10.3 ± 0.2^{b}	8.4 ± 0.2^{a}	145 ± 1.9^{a}
IRD	5.6 ± 0.1^{ef}	4.5 ± 0.1^{d}	45 ± 0.7^{ef}
VD	5.4 ± 0.1^{f}	4.4 ± 0.1^{d}	40 ± 0.5^{g}
4min-IRD-FD	7.3 ± 0.2^{d}	5.8 ± 0.1^{b}	59 ± 0.9^{d}
6min-IRD-FD	11.5 ± 0.2^{a}	8.7 ± 0.2^{a}	91 ± 1.3^{b}
2h-VD-FD	8.5 ± 0.1^{c}	6.1 ± 0.1^{b}	72 ± 1.1^{c}
3h-VD-FD	6.0 ± 0.1^{e}	5.1 ± 0.1^{c}	49 ± 0.8^{e}

FD: freeze drying; IRD: infrared drying; VD: vacuum drying; IRD-FD: infrared-freeze drying; VD-FD: vacuum pre- and freeze finish drying; TE: Trolox equivalents; GAE: gallic acid equivalents.
Data are expressed as the average ± standard deviation for three replicates. Values in the same column with different superscripts (a, b, c, d, e, f) are significantly different ($P \leq 0.05$).

The 6min-IRD-FD, FD, 2h-VD-FD, 4min-IRD-FD, 3h-VD-FD, IRD, and VD dried apple dices contained 8.7, 8.4, 6.1, 5.8, 5.1, 4.5, and 4.4 mg GAE/g total phenolic content on dry weight basis, respectively. Maximum retention of TPC was observed in the 6min-IRD-FD samples, but no significant difference ($P > 0.05$) was found between FD and 6min-IRD-FD apples. No significant difference ($P > 0.05$) was found for TPC retention in apple products dried by 4min-IRD-FD and 2h-VD-FD methods. The 6min-IRD-FD apple cubes had the highest TPC and AA retention among all drying modes, which is due to the shorter drying time and the shorter exposure of the sample to oxygen. The drying temperature used also has a significant effect on the antioxidant activity and polyphenol content of apples. Demiray et al. (2023) found that higher antioxidant activity and phenolic content were found in apples when hot air drying at 45 °C compared to 55 and 65 °C. Our results showed that lyophilisation also had a beneficial impact on the polyphenol and antioxidant activity of apples.

Table 3 shows the ascorbic acid (vitamin C) contents of dried apple with the single-stage and two-stage methods of drying. The FD, 6min-IRD-FD, 2h-VD-FD, 4min-IRD-FD, 3h-VD-FD, IRD, and VD dried apple cubes contained 145, 91, 72, 59, 49, 45, and 40 mg/100 g ascorbic acid content on dry weight basis, respectively. Retention of ascorbic acid in apple was significantly higher ($P < 0$ 0.05) in FD drying as compared to hybrid dried samples. According to Shewale and Hebbar (2017), the absence of air and very low drying temperatures typical of lyophilisation can cause vitamin C and phenolics retention.

Among the combined methods, apple dried by the 6min-IRD-FD method had the highest ($P < 0.05$) vitamin C content. 6min-IR-FD dried apple retained 62.7% of the ascorbic acid content compared to FD, which could be attributed to quicker pre-drying performed at lower temperature (40 °C). Both single-stage dehydration methods resulted in significant loss of vitamin C during drying, but the infrared dried sample showed less loss of ascorbic acid ($P < 0.05$) than the vacuum dried sample. The reduction in heat-sensitive vitamin C can also be due to the length of time required for drying in VD-FD. In the present study, about 50–66% loss of ascorbic acid was observed in VD-FD dried apple cubes compared to FD.

4. CONCLUSIONS

In this study, the results of single-stage and two-stage drying of apple cubes were reported in terms of drying time, water activity, rehydration, texture, antioxidant activity, phenolic content, and ascorbic acid content. Depending on drying programs and methods, combined drying (IRD-FD and VD-FD) resulted in a reduction in the drying time to an extent of 31.8–49.5% in comparison to freeze drying. Considering the water activity value, apple cubes preserved by all drying methods were microbiologically stable. The increase in pre-drying time for IRD and VD in combined drying had no relevant effect on the physical characteristics of the apple product. The total polyphenol, antioxidant activity, and vitamin C values improved with the rise of pre-drying time (from 4 to 6 min) at infrared-assisted freeze drying. The dried apple cubes had slightly higher antioxidant activity, polyphenols content, ascorbic acid content, hardness parameter, and same rehydration capacity and water activity when dried by infrared-assisted freeze drying as compared to the vacuum drying-assisted freeze drying. Compared to freeze drying, the freeze drying combined with infrared drying achieved similar or better product quality, in particular in rehydration, vitamin C content, antioxidant activity, and polyphenol content.

We concluded that 6min-IRD-FD (infrared drying-assisted freeze drying) with a drying temperature of 40 °C was the optimal solution among the investigated one- and two-stage drying methods in case of apple with respect to drying time, rehydration, texture, water activity, and bioactive components.

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