Effect of freeze drying, hot air drying, and hot air drying preceded by freezing on phytochemical composition, antioxidant activity, and technological properties of mango peels

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10 **Tables**

Table 1. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on mango peels' nutritional composition.

	Dry Weight g/ 100 g FW	Total Carbohydrates g/ 100 g DW	Total fiber g/ 100 g DW	Soluble fiber g/ 100 g DW	Insoluble fiber g/ 100 g DW	Protein g/ 100 g DW	Fat g/ 100 g DW	Ash g/ 100 g DW	Vitamin C mg/ 100 g DW
FS	16.83±0.68^{B}	90.15±0.54^{B}	33.62±1.43^{A*}	16.57±0.60^{A*}	17.69±0.33^{A*}	5.88±0.74^{A}	1.92±0.05^{A*}	2.05±0.34^{B}	488.22±20.49^{A}
FD	94.37±0.46^{A}	90.63±0.36^{AB}	33.31±0.96^{A}	15.57±0.76^{A}	17.44±1.10^{A}	4.96±0.33^{A}	1.89±0.04^{A}	2.53±0.00^{AB}	442.49±7.57^{B}
HAD	90.83±3.27^{A}	90.24±0.24^{B}	33.62±1.43^{A*}	16.57±0.60^{A*}	17.69±0.33^{A*}	4.95±0.08^{A}	1.92±0.05^{A*}	2.89±0.20^{A}	221.62±2.62^{C}
FZ+HAD	93.47±0.78^{A}	91.26±0.26^{A}	33.09±1.10^{A}	16.41±1.06^{A}	17.69±0.11^{A}	4.81±0.22^{A}	1.56±0.02^{B}	2.36±0.03^{B}	134.74±1.90^{D}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). *The soluble, insoluble, and total fiber contents and fat amount in fresh mango peels were estimated using the HAD sample since the methodologies applied to quantify these nutrients are inadequate for fresh samples. FW – fresh weight; DW – dry weight; FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 2. Composition of soluble and insoluble fiber from freeze dried, hot air dried, and frozen and hot air-dried mango peels.

		Acid insoluble lignin and protein g/ 100 g of fiber	Uronic Acids g/ 100 g of fiber	Glucose g/ 100 of fiber	Galactose g/ 100 g of fiber	Xylose g/ 100 g of fiber	Arabinose g/ 100 g of fiber
Soluble Fiber	**FD**	-	39.87±1.97^{A}	4.31±0.08^{B}	21.08±0.82^{A}	nd	17.58±1.18^{A}
	HAD	-	42.55±1.34^{A}	3.31±0.05^{C}	21.11±0.29^{A}	nd	16.86±0.77^{A}
	FZ+HAD	-	38.22±1.92^{A}	6.31±0.18^{A}	20.38±0.78^{A}	nd	17.47±0.30^{A}
Insoluble fiber	**FD**	16.73±1.17^{A}	10.70±0.44^{C}	33.50±0.38^{C}	8.09±1.33^{A}	5.83±0.07^{A}	2.24±0.26^{A}
	HAD	15.72±0.75^{A}	14.15±0.31^{A}	36.34±0.41^{B}	7.30±0.82^{A}	5.88±0.12^{A}	2.17±0.54^{A}
	FZ+HAD	15.97±0.76^{A}	12.96±0.57^{B}	39.03±0.01^{A}	6.95±0.49^{A}	6.34±0.39^{A}	2.44±0.82^{A}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h); nd – not detected.

Table 3. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on free and bound phenolic compounds released from mango peels through methanolic extraction, basic and acid hydrolyses, respectively.

	Methanolic extraction µg / g DW				Basic hydrolysis µg / g DW				Acid hydrolysis µg / g DW			
	FS	**FD**	**HAD**	**FZ+HAD**	**FS**	**FD**	**HAD**	**FZ+HAD**	**FS**	**FD**	**HAD**	**FZ+HAD**
Total phenolic compounds	12383.18±2147.47^{A}	10624.73±1392.74^{A,B}	7680.63±103.33^{B,C}	6313.85±333.03^{C}	1075.56±270.66^{A}	314.40±41.88^{B}	631.06±46.08^{B}	644.95±17.84^{B}	221.95±11.47^{A}	68.73±11.01^{C}	113.56±12.62^{B}	120.91±20.60^{B}
Mangiferin	963.77±116.65^{A}	863.50±10.66^{A}	632.50±10.11^{B}	443.81±3.26^{C}	34.35±5.84^{A}	9.51±2.87^{B}	28.84±6.85^{A}	22.87±6.61^{A}	9.21±1.33^{A}	2.18±0.52^{B}	6.92±1.50^{A}	6.25±1.45^{A}
Gallic acid	610.68±52.39^{B}	650.32±20.53^{B}	830.29±9.07^{A}	508.87±8.96^{C}	533.04±101.22^{A}	42.65±1.63^{C}	152.66±26.58^{BC}	207.74±7.32^{B}	92.81±25.13^{A}	12.27±0.27^{B}	23.25±0.03^{B}	28.26±1.21^{B}
Penta-O-galloyl-β-D-glucose	401.17±50.00^{A}	384.21±9.76^{A}	358.72±13.26^{A}	160.83±50.81^{B}	nd	nd	nd	nd	nd	nd	nd	nd
Methyl gallate	323.74±53.86^{A}	299.31±2.22^{A}	188.72±13.61^{B}	93.38±5.49^{C}	nd	nd	nd	nd	nd	nd	nd	nd
Catechin	409.16±19.86^{B}	485.27±6.62^{A}	249.03±15.98^{C}	118.00±14.15^{D}	nd	nd	nd	nd	nd	nd	nd	nd
Quercetin-3-O-galactoside[a]	490.95±65.86^{A}	464.20±3.42^{A,B}	381.88±0.79^{B}	513.04±31.56^{A}	19.99±1.74^{A}	8.72±1.64^{C}	18.71±1.72^{AB}	14.54±3.24^{B}	nd	nd	nd	nd
Quercetin 3-β-D-glucoside	183.09±16.69^{A}	185.26±0.85^{A}	144.87±0.77^{B}	138.93±7.56^{B}	20.53±4.39^{A}	7.97±1.80^{B}	20.38±2.05^{A}	15.76±0.39^{A}	nd	nd	nd	nd
Quercetin 3-O-α-L-arabinopyranoside	79.17±11.46^{A}	80.08±0.74^{A}	63.84±0.96^{B}	53.08±2.02^{B}	7.56±1.54^{A}	3.54±0.01^{B}	7.71±0.69^{A}	4.65±1.44^{B}	nd	nd	nd	nd
Quercetin-3-O-α-L-arabinofuranoside	52.05±7.71^{A}	50.59±0.16^{A}	39.70±0.79^{B}	35.49±1.54^{B}	4.89±1.00^{A}	2.29±0.36^{B}	4.71±0.32^{A}	3.18±0.92^{AB}	nd	nd	nd	nd
Ferulic acid	nd	nd	nd	nd	6.26±1.32^{B}	3.45±0.37^{C}	8.81±0.65^{A}	6.48±0.08^{B}	nd	nd	nd	nd
p-Coumaric acid	nd	nd	nd	nd	13.29±0.04^{A}	10.18±0.24^{B}	14.12±1.27^{A}	11.24±0.13^{B}	2.01±0.21^{A}	1.38±0.15^{B}	2.04±0.21^{A}	1.27±0.09^{B}
3,4-dihydroxybenzoic acid	nd	nd	nd	nd	47.59±3.43^{B}	52.72±8.84^{B}	81.82±14.95^{A}	23.23±0.02^{C}	nd	nd	nd	nd
4-hydroxybenzoic acid	nd	nd	nd	nd	66.26±9.69^{A}	16.69±1.20^{C}	50.39±7.23^{A,B}	46.15±10.79^{B}	25.86±1.48^{A}	2.58±0.25^{C}	6.12±0.43^{B}	5.63±0.87^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same row indicate statistically significant differences between samples ($p < 0.05$). [a]In HPLC method used quercetin-3-O-galactoside and ellagic acid had the same retention time, so these values can correspond to these both compounds. DW – dry weight; nd –

not detected; FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

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41 **Table 4.** Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on mango peels' carotenoids.

	β-carotene µg/ g DW	Lutein[a] µg/ g DW	Violaxanthin µg/ g DW
FS	388.97±11.56^{A}	15.12±1.14^{B}	8.47±0.11^{A}
FD	396.77±34.59^{A}	16.16±1.60^{AB}	4.28±0.84^{B}
HAD	429.25±20.38^{A}	18.96±1.46^{A}	1.11±0.09^{C}
FZ+HAD	249.91±32.64^{B}	11.76±1.35^{C}	nd

42 Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$).
43 [a]In the HPLC method lutein and zeaxanthin had the same retention time, so these values can correspond to these both compounds. DW – dry weight; nd – not detected; FS –
44 fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air
45 drying (at 65 ºC for 48 h).

Table 5. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on antioxidant activity of free and bound phenolic compounds released from mango peels through methanolic extraction and basic and acid hydrolyses, respectively.

		ABTS	DPPH
		μg of Trolox eq. / g DW	μg of Trolox eq. / g DW
Free phenolic compounds	**FS**	30567.99±1940.59^{A}	28429.99±3251.63^{A}
	FD	27262.52±1865.66^{A}	24766.32±1729.97^{A}
	HAD	17384.16±697.05^{B}	14717.43±1352.84^{B}
	FZ+HAD	16804.20±2292.16^{B}	13706.52±2313.19^{B}
Bound phenolic compounds (basic hydrolysis)	**FS**	3259.78±809.41^{A}	2591.03±757.94^{A}
	FD	621.56±28.51^{B}	390.29±32.31^{C}
	HAD	1364.10±117.82^{B}	1153.44±109.63^{BC}
	FZ+HAD	1622.91±57.97^{B}	1380.51±31.00^{B}
Bound phenolic compounds (acid hydrolysis)	**FS**	510.02±92.15^{A}	346.81±51.87^{A}
	FD	128.20±14.41^{B}	72.33±14.02^{C}
	HAD	217.18±37.06^{B}	179.54±8.71^{B}
	FZ+HAD	258.42±55.16^{B}	214.32±55.81^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 6. Particle size distribution in freeze dried, hot air-dried, and frozen and hot air-dried samples.

	>500 µm g/ 100 g	250 – 500 µm g/ 100 g	150 – 250 µm g/ 100 g	100 – 150 µm g/ 100 g	50 – 100 µm g/ 100 g	<50 µm g/ 100 g
FD	6.03±0.29^{C}	11.79±0.16^{C}	13.92±0.97^{B}	12.62±1.20^{A}	31.15±4.95^{A}	24.49±4.33^{A}
HAD	18.26±1.28^{A}	26.88±0.96^{B}	17.22±1.06^{A}	10.90±0.45^{A}	15.70±1.58^{B}	11.03±1.61^{B}
FZ+HAD	14.75±0.41^{B}	29.16±0.67^{A}	18.71±0.07^{A}	12.48±0.48^{A}	15.46±0.88^{B}	9.45±1.31^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). DW – dry weight; W – washed sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 7. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on mango peel powders' color, water absorption capacity, and oil absorption capacity.

	Color		Water Absorption Capacity	Oil Absorption Capacity
	L*	Hue Angle	g of water/ g of sample	g of oil/ g of sample
FD	20.21±0.46^{A}	89.54±0.01^{A}	5.04±0.07^{B}	2.03±0.11^{A}
HAD	15.62±0.23^{B}	81.42±0.20^{B}	6.29±0.25^{A}	1.40±0.07^{B}
FZ+HAD	14.39±0.41^{C}	77.65±1.15^{C}	6.23±0.37^{A}	1.28±0.15^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

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This study evaluated the impact of freeze drying (FD), hot air-drying (65 ºC; 48 h) (HAD), and HAD preceded by freezing (-20 ºC; 30 days) (FZ+HAD) on mango peels' composition, antioxidant activity, and technological properties. Overall, drying had no marked effect on macronutrients and fiber composition. FD (-9%), HAD (-55%), and FZ+HAD (-72%) reduced vitamin C. FD preserved all free phenolics, while HAD (-20 – -42%) and FZ+HAD (-17 – -71%) decreased most of them. HAD, FZ+HAD, and FD reduced 2, 6, and 9 of the 10 bound phenolics identified. FZ+HAD performed worst in preserving β-carotene, lutein, and violaxanthin. All drying methods impaired bound phenolics' antioxidant activity. FZ+HAD was the darker powder. FD showed the best and worst oil and water absorption capacities. This study evaluated the impact of drying on mango peels' bound phenolics and compared HAD with FZ+HAD for the first time. FZ+HAD was inadequate regarding antioxidants preservation.

Keywords: mango byproducts; drying; fiber; free and bound phenolics; carotenoids; antioxidant activity; technological properties

1. Introduction

A tremendous amount of mango peels (7 – 9 million tons) are discarded yearly (Marçal et al., 2024). The deposition of fruit and vegetable byproducts in landfills or their incineration causes severe environmental problems, namely greenhouse gas emissions, water and soil pollution, and eutrophication (Kučuk et al., 2024; Leong & Chang, 2022).

During the last two decades, several research works (>270 papers available in the Web of Science) were performed to determine mango peels' nutritional composition, bioactive compounds profile, and health and technological benefits (Marçal & Pintado, 2021; Tariq et al., 2023). Nowadays, it is consensual among the scientific community that mango peels have great potential for use in the food industry as a functional ingredient or as a source of natural additives due to their high amounts of fiber, vitamin C, phenolic compounds (mostly xanthones, flavonoids, gallic acid and its derivatives), and carotenoids, which ascribe their antioxidant, antimicrobial, anti-diabetic, anti-inflammatory and prebiotic activities (Gupta et al., 2022; Kučuk et al., 2024; Marçal & Pintado, 2021).

The valorization of mango peels in the food industry possesses health, environmental, and economic advantages (Antoniolli et al., 2023). In addition to their nutritional value and health benefits aforementioned, mango peels' use contributes to solving the environmental and economic problems related to discard, prevents more natural resources from being spent to produce the ingredients or additives they replace, and can represent a new source of financial income for mango processing companies (Matharu et al., 2016). However, the valorization of mango peels had some challenges that need to be overcome, namely, their high perishability, seasonality, and chemical composition variability (Antoniolli et al., 2023; Dukare et al., 2022; Marçal et al., 2024).

Drying has been widely used to preserve mango peels, other fruit byproducts, as well as edible fruits and vegetables (Athira et al., 2023; Ma et al., 2023; Marçal & Pintado, 2021; Tariq et al., 2023; Zuñiga-Martínez et al., 2022). Besides extending mango peels' shelf-life due to reducing spoilage microorganisms and enzymes' activity, drying decreases peels' weight and volume, facilitating transportation and storage (Dukare et al., 2022; Ma et al., 2023). Moreover, processing mango peels into powders makes their introduction into some kinds of foods more convenient (e.g., bakery products, pasta, and breakfast cereals) (Ma et al., 2023; Marçal & Pintado, 2021; Zuñiga-Martínez et al., 2022). However, drying modifies mango peels' phytochemical composition, bioactivity, and sensory properties. The extent of these changes is strongly affected by drying conditions (temperature, time, process), the kind of pretreatment applied (e.g., blanching, irradiation, pulsed electric field, and pressing), and mango peels' characteristics (e.g., cultivar, ripeness stage, and postharvest conditions) (Geerkens, Miller-Rostek, et al., 2015; Geerkens, Nagel, et al., 2015; Nagel et al., 2014; Palmeira et al., 2012). Therefore, the drying conditions and their combination with pretreatment should be selected considering the initial characteristics of mango peels and the desired final product, without forgetting economic and environmental sustainability.

A search on Web of Science, in which all papers with the words "drying" in the title/ abstract/ keywords and "mango peels or mango byproducts/ by-products" in any field of the manuscript were analyzed, showed that 22 research studies have been published encompassing the effect of drying on mango peels' composition and properties since 1991 up to April 2024. Only experimental papers in English that compare fresh mango peels with dried ones, different drying methods, or distinct drying conditions were considered. These 22 research works studied the following drying methods: intermittent microwave-convective drying (Nagel et al., 2017); drum drying (Antoniolli et al., 2023; Troiani et al., 2022); vacuum drying at 60 ºC (Sogi et al., 2013); infra-red drying (Sogi et al., 2013); freeze drying

(FD) (Ancos et al., 2018; Berardini et al., 2005; del Pilar Sanchez-Camargo et al., 2019; Dorta et al., 2012; Garcia-Amezquita et al., 2018; Geerkens, Miller-Rostek, et al., 2015; Geerkens, Nagel, et al., 2015; Oliver-Simancas et al., 2020; Sogi et al., 2013); air drying at room temperature (Kučuk et al., 2024); solar drying (Geerkens, Miller-Rostek, et al., 2015) hot air drying in fluidized-bed at 50 - 80 ºC (Geerkens, Miller-Rostek, et al., 2015; Nagel et al., 2014, 2017); and hot air drying in oven (HAD) at 45 – 100 ºC (Ancos et al., 2018; Berardini et al., 2005; de Brito Araújo et al., 2021; del Pilar Sanchez-Camargo et al., 2019; Dorta et al., 2012; Dukare et al., 2022; Garcia-Amezquita et al., 2018; Geerkens, Miller-Rostek, et al., 2015; Oliver-Simancas et al., 2020; Roa-Tort et al., 2024; Sant'Anna et al., 2014; Sogi et al., 2013; Zulkifli et al., 2012). Additionally, some studies evaluated the combination of pretreatments with HAD, namely pulse electric field (Santos et al., 2023), blanching (Geerkens, Nagel, et al., 2015), and washing with ethanol (Kratchanova et al., 1991). Finally, (Palmeira et al., 2012) did not specify the drying method used.

The HAD has been the most studied method for drying mango peels, followed by FD. This may be because HAD is simultaneously among the simpler, cheaper, and faster methods, while FD is the golden standard for preserving thermolabile compounds (Ancos et al., 2018; del Pilar Sanchez-Camargo et al., 2019). However, some lack of knowledge was identified, namely the effect of HAD, FD, and any other drying method on mango peels' bound phenolic compounds. Studies with other raw materials observed that drying conditions highly affect the bound phenolics content. For instance, Lang et al. (2019) showed that the bound phenolics content in HAD black rice varied depending on drying temperature, while Ma et al. (2023) reported that FD usually converts part of bound phenolic compounds into free ones. Phenolic compounds are in free or bound form, highly influencing their bioaccessibility and, consequently, their health benefits in the human body (Acosta-Estrada et al., 2014).

Furthermore, although in some research works, the mango peels were frozen before they were submitted to HAD (Berardini et al., 2005; Dorta et al., 2012; Palmeira et al., 2012), HAD of fresh mango peels was never compared with HAD preceded by freezing (FZ+HAD). So, the impact of freezing as a previous step on HAD mango peels is unknown. However, freezing can often be necessary as a complementary preserving method to HAD, either in academia or in industry, when mango peels cannot be dried during their shelf-life due to, for instance, long transport, limited drying space, the necessity of evaluating different drying conditions using the same equipment and the same mango peels batch (e.g., several days can be necessary to evaluate the impact of HAD at different temperatures in the same oven, so if the same mango peel batch is used, they need to be preserved for instance using freezing, to avoid their deterioration), among others.

Considering the lack of knowledge identified above, the present study aimed to evaluate the impact of FD, HAD (65 ºC, 48 h), and HAD (65 ºC, 48 h) preceded by freezing at -20 ºC for 30 days on mango peel powders' macronutrients, fiber composition, vitamin C, free and bound phenolic compound profiles, carotenoids, antioxidant activity, and technological properties. All these parameters were also assessed in fresh mango peels, excluding technological properties and fiber composition.

2. Materials and Methods

2.1 Chemicals

All sugars, phenolic compounds, and carotenoids standards were purchased from Sigma-Aldrich (St. Louis, USA) or Extrasynthese (Genay, France). Trifluoroacetic acid, ascorbic acid, dithiothreitol, phosphoric acid, KH2PO4, 2-azinobis-3-ethylbenzothiazoline-6-sulphonic acid (ABTS), 2,2-azo-bis-(2-methylpropionamidine)-dihydrochloride (AAPH), 2,2-diphenyl-1-picrylhydrazyl (DPPH), Folin-Ciocalteu's reagent, Trolox, 3,4-

dimethylphenol, calcium carbonate, sodium hydroxide, sulfuric acid, hydrochloric acid, and boric acid were acquired at Sigma-Aldrich (St. Louis, USA). Acetone and dichloromethane were obtained from Honeywell Fluka (North Carolina, USA). Hexane and absolute ethanol were purchased from Carlos Erba Reagents (Barcelona, Spain). Methanol was acquired at VWR Chemicals (Pennsylvania, USA). Ethyl acetate and acetonitrile were obtained from Fisher Chemical (New Hampshire, USA). Christeyns (Agualva - Cacém, Portugal) provided the commercial disinfectant with peracetic acid (Mida Chriox 5).

2.2 Raw Materials

All mango peels analyzed in this study were from the "Tommy Atkins" cultivar, cultivated in Brazil and commercialized in Portugal by the company Nuvi Fruits, SA. Peels used to perform all assays, excluding carotenoids and vitamin C, were from mangoes harvested in 2022 and mechanically peeled in Nuvi Fruits, SA. The transportation of these peels from the factory to the laboratory was done under refrigerated conditions and within 24 after the peeling process. Peels used to determine vitamin C and carotenoids profile were harvested in 2024 and manually peeled at the laboratory with a kitchen knife. The manual peeling was carried out to obtain byproducts like those resulting from the mechanical process, ensuring that the amount of mesocarp removed along with the epicarp was similar. Furthermore, after manual peeling, peels were kept under refrigerated conditions for 24 h to simulate transportation from the factory to the laboratory.

2.3 Mango peels processing and storage

First, mango peels were cut into pieces of about 5 g and mixed to ensure the similitude between samples submitted to different processing conditions. After, they were washed with peracetic acid following the conditions recommended by Marçal & Pintado (2021) (mango peels to disinfectant solution ratio: 1:1 (kg:L); peracetic acid concentration: 27 mg/L;

disinfection time: 19 min) **(fresh sample (FS))**. Then, washed samples were submitted to three different processes: FD, with a condenser temperature of -63 ºC and chamber vacuum pressure of 0.10 mbar for 96 h **(freeze-dried samples (FD)**; HAD at 65 ºC for 48 h, with constant air circulation **(hot-air dried samples (HAD))**; and HAD at 65 ºC for 48 h, with constant air circulation preceded by freezing at -20 ºC for 30 days **(Frozen and hot-air dried samples (FZ+HAD))**. In both HAD processes, the airflow and relative humidity were 1.6 m/s and 53%, respectively. Finally, dried peels were milled using a coffee grinder (TAURUS Aromatic II, Barcelona, Spain). Immediately before performing each assay, fresh peels (about 50 g) were also milled to ensure the sample's homogeneity and representativeness.

Free phenolic compounds, carotenoids, and vitamin C extractions were done right after the processing. Until the remaining assays were performed, mango peel powders were stored in a desiccator at room temperature while fresh peels were kept at –80 ºC. All samples were protected from light during all storage time.

2.4 Nutritional composition

Fresh and dried mango peels were submitted to Standard AOAC (1990) methods to quantify their dry weight (DW), protein (factor conversion: 6.25), and ash. Fat and soluble, insoluble, and total fiber (obtained as the sum of soluble and insoluble fiber) were evaluated following the procedures described by Gómez-García et al. (2021) and Megazyme (2017), respectively. Both methodologies are only adequate for dried samples, so these nutrients were only determined in mango peel powders. It was assumed that fresh peels' fat, soluble, insoluble, and total fiber were the same as those in the HAD sample. Finally, total carbohydrates were calculated by difference [100 − (% moisture + % ash + % protein + % lipid)].

Regarding data presentation, DW was expressed in g/ 100 g of fresh weight (FW), while all other nutrients were shown in g/ 100 g of DW.

2.5 Composition of soluble and insoluble fiber

2.5.1 Hydrolysis

First, the method described by Megazyme (2017) was performed to isolate soluble and insoluble fiber from the other components of mango peel powders. Then, insoluble and soluble fibers were collected from crucibles, crushed, and kept in a desiccator until the hydrolyses were done.

Two different methodologies, Sluiter et al. (2008) and Ribeiro et al. (2021), were used to hydrolysate insoluble and soluble fiber, respectively. Regarding insoluble fiber, first, 250 mg of this nutrient was mixed with 3 mL of sulfuric acid (72% w:w) and placed in a water bath at 30 ºC for 1 h. Samples were shaken in a vortex every 5 min. Afterward, 84 mL of distilled water was added to the samples, and they were autoclaved for 1 h at 121 ºC. Finally, autoclaved slurries were filtered through crucibles. The solid particles retained in crucibles were used to quantify acid-insoluble lignin, while filtrates were used to determine neutral sugar profiles and uronic acids (Sluiter et al., 2008).

Concerning soluble fiber, first, 30 mg of this nutrient and 12 mL of sulfuric acid (6% w:w) were mixed using a vortex. Then, these mixtures were autoclaved at 121 ºC for 1 h. Finally, the autoclaved solutions were used to determine neutral sugars and uronic acids (Ribeiro et al., 2021).

2.5.2 Acid insoluble lignin

The crucibles were dried until constant weight at 105 ºC before and after filtration, and the amount of ash retained in crucibles along with acid-insoluble lignin was calculated through incineration at 575 ºC for 180 min (Sluiter et al., 2008). Hence, the acid-insoluble lignin was calculated by subtracting the weight of ashes from the weight of solid material retained in crucibles. The results were expressed in g/ 100 g of fiber DW.

2.5.3 Uronic acids quantification

Uronic acids were quantified using the colorimetric assay described by Ribeiro et al. (2020). Briefly, 250 µL of hydrolyzed fiber or galacturonic acid standards (to calibration curve) was mixed with 250 µL of a boric acid and chloride acid solution, and 4 mL of sulfuric acid 96%. Then, they were incubated in a water bath at 70 ºC for 40 min. After, 200 µL of dimethylphenol were added to solutions previously cooled at room temperature. Finally, the absorbances of these solutions were measured at 400 nm and 450 nm. The results were presented in g/ 100 g of fiber DW.

2.5.4 Neutral sugar identification and quantification by HPLC

Neutral sugars were identified and quantified following the method recommended by Sluiter et al. (2008). First, the pH of solutions with hydrolyzed fiber (5 mL) was adjusted with calcium carbonate to values between 5 and 6. Then, the solutions were filtrated with 0.45 µm membrane and analyzed in a Beckman Coulter System Gold HPLC (Knauer, Berlin, Germany) coupled to an Infrared detector. The stationary phase was an Aminex HPX-87P Column (300 x 7.8 mm, 9 µm particle size) (Bio-Rad, California, USA) at 85 ºC, while the mobile phase was ultra-pure water. The injection volume, flow, and running time were 30 µL, 0.60 mL/ min, and 30 min, respectively. Identification and quantification of neutral sugars were made by comparing peaks' retention time and area with calibration curves of pure standards of glucose, xylose, galactose, arabinose, and mannose. The results were shown in g/ 100 g of fiber DW.

2.6 Vitamin C

Brause et al. (2003) described the methodology used to extract and quantify vitamin C in fresh and dried mango peels. Briefly, a solid-liquid aqueous extraction was done (1 g DW

to 40 mL of water) using a dispersing machine (IKA Ultra-turrax T18, Wilmington, USA) at 19 000 rpm for 1 min. Then, the extracts were separated from the residue by centrifugation (3900 *g*, 4ºC, 20 min). Immediately after centrifugation, 1 mg of dithiothreitol was added per mL of extracts, and they were mixed for 2 h in a roller tube at room temperature. Finally, extracts were filtrated with 0.22 µm membrane, and vitamin C was quantified by HPLC using the same equipment and methodology described in Marçal et al. (2022). Throughout the process, samples/ extracts were always protected from light. The results were expressed in mg/ 100 g of mango peels DW.

2.7 Phenolic compounds

2.7.1 Methanolic extraction of free phenolic compounds

Extracts with free phenolic compounds were produced, according to Marçal et al. (2022). First, a solid-liquid methanolic extraction was carried out (1 g of DW to 25 mL of methanol 80%) using a dispersing machine at 24 000 rpm for 1 min. Then, a centrifugation (5 000 rpm, 20 min, 4 ºC) was performed to separate the extracts containing free phenolic compounds **(FPC)** from the solid residues (pellet) with bound (non-extractable) phenolic compounds linked.

The extracts **(FPC)** were stored at -80 ºC until they were analyzed, while residues were kept at -20 ºC until the hydrolyses of bound phenolic compounds were performed.

2.7.2 Basic and acid hydrolyses of bound phenolic compounds

Extracts with bound phenolic compounds were obtained following the procedure described by Marçal et al. (2024) and using the same equipment. Briefly, bound phenolic compounds were released from residues (pellets) of methanolic extraction through a basic hydrolysis with sodium hydroxide 4 M for 4 h, at 22 ºC and 250 rpm, followed by an acid hydrolysis with HCl 2 M at 85 ºC for 1 h. Then, the pH of each hydrolysis supernatant was

adjusted to 2, and they were extracted thrice with ethyl acetate. Finally, ethyl acetate was totally evaporated at 30 ºC, and dried bound phenolic compounds from basic (BBPC) and acid hydrolyses (ABPC) were dissolved in 2 mL of methanol.

2.7.3 Total phenolic compounds

Folin–Ciocalteu method was carried out to quantify total phenolic compounds in the three phenolic extracts of each sample (FPC, BBPC, and ABPC). This methodology was performed according to Vilas-Boas et al. (2020). The results were shown in μg of gallic acid equivalents (GAEs)/ g of mango peels DW.

2.7.4 Identification and quantification by HPLC

Identification and quantification of main free and bound phenolic compounds were performed by HPLC using the same procedure and equipment described in Marçal et al. (2024). The results were shown in μg/ g of mango peels DW.

2.8 Carotenoids

2.8.1 Extraction

The extraction of carotenoids was done according to Stinco et al. (2014) and Marçal et al. (2022) with some modifications. Briefly, carotenoids from 2.15 g of fresh mango peels or 0.40 g of mango peel powders were extracted with 5 mL of a hexane: acetone solution (extraction conditions: 1 g of mango peels DW: 12.50 mL of acetone: hexane (1:1; v/v)), using a dispersing machine for 30 s at 19 000 rpm. After, the slurries were centrifuged for 15 min at 4 ºC and 3 900 *g* and the supernatants were collected. The extraction was repeated until the residues were colorless. Then, the supernatants from the successive extractions were concentrated using an RVC 2–18 speed-vacuum evaporator (Christ, Osterode am Harz, Germany) for 30 min at 30 °C. Finally, saponification was performed by mixing concentrated

extracts with 4 mL of methanolic KOH (30%; m/v) and 4 mL of dichloromethane in a tube roller for 1 h at room temperature. After removing KOH, the extracts were washed three times with ultra-pure water, and the dichloromethane evaporated using a nitrogen flow. Dried extracts were resuspended in 1.20 mL of acetone: hexane (1:1; v/v). Throughout the process, samples/ extracts were always protected from light.

2.8.2 Identification and quantification by HPLC

Carotenoids were identified and quantified by HPLC using the same methodology and equipment as Marçal et al. (2024). The results were shown in μg/ g of mango peels DW.

2.9 Antioxidant activity

The capacity of phenolic extracts to scavenge the ABTS and DPPH-free radicals was evaluated following the procedures described by Vilas-Boas et al. (2020). The results were depicted in μg of Trolox equivalents (TEs)/ g of mango peels DW.

2.10 Technological Properties

Water and oil absorption capacities were evaluated following the methodologies used by Chandra et al. (2015), with slight modifications. First, 1 g of mango peel powders were mixed with 10 mL of deionized water or 10 mL of vegetal oil and kept in a water bath at 60 ºC for 30 min. Then, the slurries were centrifuged at 22 ºC and 2 200 *g* for 15 min. The results were expressed in g of water or oil/ g of powders DW.

Regarding mango peel powders' color, D65 illuminant and the CIE (Commission Internationale de l'Eclairage) parameters (L*, a*, b*) were measured using a CR-400 colorimeter (Konica Minolta, Osaka, Japan). Results were presented as hue angle ($h^{\circ} = \arctan(b^*/a^*)$).

The particle size distribution of mango peel powders was determined using RETSCH woven wire mesh sieves and a Horizontal Sieve Shaker AS 200 Control (RETSCH, Germany) at an amplitude of 100% for 20 min. The results were presented in g/ 100 g of mango peel powders.

2.11 Statistical analysis

Before analyzing and presenting the results, all of them (excluding DW) were converted into 100% DW to offset the variation due to moisture.

The statistical analysis was performed with IMB SPSS Statistics Software (New York, USA). First, the normality of the data distribution was verified using the Shapiro-Wilk test. Then, ANOVA one-way and Turkey's post hoc tests were carried out ($p < 0.05$).

3. Results

3.1 Nutritional composition

Table 1 depicts the nutritional composition of fresh and dried mango peels. Fresh mango peels were mainly composed of water, but as expected, all drying methods reduced the moisture content to less than 10 g/ 100 g FW. Hence, the main constituent of the three mango peel powders was carbohydrates (> 90 g/ 100 g DW). The FZ+HAD sample showed a slight increase in total carbohydrate content (+1%) comparing fresh mango peels and other powders. In all samples, about 37% of total carbohydrates corresponded to dietary fiber. No differences were detected between the soluble, insoluble, and total fiber contents in FD, HAD, and FZ+HAD mango peel powders. The protein content was also similar in all samples. Analyzing fat content, it was observed that the FZ+HAD sample showed less 18.11% of this nutrient than the other powders.

Regarding the impact of drying on micronutrients, results showed that FD, HAD, and FZ+HAD did not cause ash reductions; however, they all significantly reduced vitamin C

316 content. FD showed the best performance regarding preserving this micronutrient, followed
317 by HAD and FZ+HAD, since they led to a decrease of 9.37%, 54.61%, and 72.40%,
318 respectively, compared with the fresh sample.

319 **3.2 Composition of soluble and insoluble fiber**

320 Table 2 shows the soluble and insoluble fiber composition of mango peel powders.
321 Uronic acids were the most abundant component in soluble fiber for the three powders,
322 followed by galactose, arabinose, and glucose. No differences were detected between the
323 components of soluble fiber among all samples, excluding glucose, which was higher in
324 FZ+HAD (+46.40% and +90.63% than in FD and HAD, respectively).

325 Regarding insoluble fiber, the main constituent in the three powders was glucose,
326 followed by acid-insoluble lignin, uronic acids, galactose, xylose, and arabinose. The acid-
327 insoluble lignin content can be slightly overestimated, as discussed in section 4.1. Insoluble
328 fiber from FD, HAD, and FZ+HAD powders had different contents of glucose and uronic
329 acids, while the other constituents were similar in all samples. The highest glucose and uronic
330 acid contents were detected in FZ+HAD (+7.40% and +16.50% than in HAD and FD) and
331 HAD (+9.18% and +32.24% than in FZ+HAD and FD) samples, respectively.

332 **3.3 Phenolic compounds**

333 Table 3 displays the free and bound phenolic compounds released from mango peels
334 through methanolic extraction and basic and acid hydrolyses, respectively. Thirteen phenolic
335 compounds were identified in all samples. Regarding free phenolic compounds, the main
336 ones in FS, FD, and HAD samples were mangiferin and gallic acid, while in FZ+HAD
337 powder, the most abundant ones were quercetin-3-O-galactoside and gallic acid. The peak
338 identified and quantified as quercetin-3-O-galactoside can also correspond to ellagic acid
339 since they have the same retention time in the HPLC method used. Besides these, the other

six compounds belonging to flavonoids and gallates families were also identified as free phenolics.

Regarding bound phenolics, data showed that basic hydrolysis resulted in extracts with a higher diversity of compounds than acid hydrolysis since in BBPC extracts, ten phenolic compounds were identified, while in ABPC extracts, only four were detected. Gallic acid was the most abundant compound in both extracts (BBPC and ABPC) from all samples, excluding the BBPC extract from FD, since this powder's main bound phenolic compound was 3,4-dihydroxybenzoic acid. Like in FPC extracts, mangiferin (xanthone) and four different quercetins (flavonoids) were detected in both bound phenolics extracts or only in BBPC extract, respectively. Furthermore, BBPC and ABPC extracts comprised phenolic compounds belonging to cinnamic acids and other phenolic acids families.

No drying methods affected the diversity of free and bound phenolic compounds. However, they markedly changed these bioactive compounds' total and individual amounts. Regarding free phenolic compounds, FD showed the best performance since, compared with the fresh sample, it did not reduce the total or any individual phenolic compound. Moreover, FD samples presented the highest amount of catechin (+18.60%, +94.86%, and +311.25% compared to FS, HAD, and FZ+HAD samples, respectively). On the other hand, HAD decreased the amount of all free phenolic compounds, excluding penta-O-galloyl-β-D-glucose and gallic acid that were preserved or increased (+35.96% than the fresh sample), respectively. Methyl gallate and catechin were the free phenolic compounds most degraded and consequently decreased by HAD (-41.71% and -39.14% than in FS sample, respectively), followed by mangiferin (-34.37% than in FS sample) and the four quercetins identified (c.a. -20% than in FS sample). Finally, FZ+HAD showed the worst performance considering the preservation of free phenolic compounds. Compared with the FS sample, FZ+HAD decreased methyl gallate and catechin 71.16%, penta-O-galloyl-β-D-glucose 59.91%, mangiferin

53.95%, and gallic acid 16.67%, while the reduction in quercetins, excluding quercetin-3-O-galactoside, were similar like those observed in HAD powders.

The three drying methods decreased the total bound phenolic compounds released through basic and acid hydrolyses. Compared with the FS sample, HAD reduced gallic acid by 71.36% and 74.95% in BBPC and ABPC extracts, respectively, and 4-hydroxybenzoic acid by 76.33% in the ABPC extract. On the other hand, the ferulic acid and 3,4-dihydroxybenzoic acid contents were 40.73% and 71.93% higher in HAD than in the FS sample, respectively, while the other phenolic compounds had similar amounts in both samples. Unlike HAD, comparing with fresh sample, FZ+HAD reduced quercetin-3-O-galactoside (-27.26%), quercetin 3-O-α-L-arabinopyranoside (-38.49%), p-coumaric acid (BBPC: -15.43%; ABPC: -36.82%), and 3,4-dihydroxybenzoic acid (-51.19%), significantly, and it did not increase ferulic acid. Like HAD, FZ+HAD also decreased gallic acid and 4-hydroxybenzoic but, in the last one, FZ+HAD caused reductions in both extracts (BBPC: -30.35%; ABPC: -78%). The amounts of the remaining three bound phenolic compounds (mangiferin, quercetin 3-β-D-glucoside, and quercetin-3-O-α-L-arabinofuranoside) were similar in FS, HAD, and FZ+HAD samples.

Finally, FD powder was the worst source of bound phenolic compounds since, compared with the FS sample, it caused drastic reductions in the contents of all bound phenolic compounds, excluding 3,4-dihydroxybenzoic acid. Furthermore, five phenolic compounds (mangiferin, quercetin-3-O-galactoside, quercetin 3-β-D-glucoside, ferulic acid, 4-hydroxybenzoic acid) showed contents significantly lower in the FD sample than in the other two powders. The lowest reductions were observed in *p*-coumaric acid (BBPC: -23.40%; ABPC: -31.34%), followed by ferulic acid (-44.89%) and the four quercetins in which the decreases varied between 53.17% and 61.18%, comparing with FS sample. The

reductions in the remaining phenolic compounds ranged between 72.31% (mangiferin in BBPC) and 92.00% (gallic acid in BBPC).

3.4 Carotenoids

Table 4 presents the carotenoids found in mango peels. β-carotene was the most abundant carotenoid in all samples, followed by lutein and violaxanthin. The peak identified and quantified as lutein can also correspond to zeaxanthin, as discussed in subsection 4.2. The three carotenoids showed different susceptibility to drying processes. β-carotene content was unaffected by FD and HAD, while FZ+HAD decreased it 35.75% compared with the fresh sample. Lutein was the only carotenoid that increased after a drying process. Its content was 25.40% higher in HAD powers than in the FS sample. On the other hand, FD caused no changes in lutein amount, while FZ+HAD reduced it by 22.22%. Violaxanthin was the most susceptible carotenoid to drying processes since its amount in FD and HAD mango peels was 49.47% and 89.89% lower than FS samples, respectively, and it was not detected in FZ+HAD powder.

3.5 Antioxidant activity

Table 5 shows the antioxidant activity of the free and bound phenolic compounds, measured through ABTS and DPPH assays. Regarding extracts with free phenolic compounds, no differences were detected between FS and FD samples in both assays. On the other hand, HAD decreased the capacities to scavenge the ABTS and DPPH-free radicals by 43.13% and 48.23%, while FZ+HAD reduced them by 45.03% and 51.79%, respectively, compared with the FS sample.

All drying methods decreased the antioxidant activity of both extracts with bound phenolic compounds (BBPC and ABPC). Concerning the capacity to scavenge DPPH-free radicals, BBPC and ABPC from FD powders showed the highest reductions (BBPC: -84.94%

and ABPC: -79.17%) compared with FS mango peels. However, HAD (BBPC: -59.35% and ABPC: -48.23%) and FZ+HAD (BBPC: -46.72% and ABPC: -38.20%) also reduced it significantly. Regarding the capacity to scavenge ABTS-free radicals, in BBPC extracts from FD, HAD, and FZ+HAD, the reductions compared with FS samples were 80.93%, 58.15%, and 50.21%, while in ABPC, they were 74.86%, 57.42%, and 49.33%, respectively. No statistically significant differences between the three powders were found in the ABTS assay.

3.6 Technological Properties

Table 6 presents the particle size distribution in the three powders. Excluding the ranges > 500 μm and 250 – 500 μm, where these samples differed by 3.51 and 2.28 g/ 100 g, respectively, HAD and FZ+HAD powders had a similar particle size distribution. On the other hand, FD powder had a significantly higher percentage of particles within the smallest ranges measured (50 – 100 μm and < 50 μm) compared with HAD and FZ+HAD samples.

Table 7 displays the technological properties, namely the water and oil absorption capacities and the color components of the three powders. The FD powder showed a lower capacity to absorb water than the HAD and FZ+HAD samples (-19.87% and -19.10%, respectively). On the other hand, FD powder had the highest oil absorption capacity, +45.00%, and +58.59%, compared to the HAD and FZ+HAD samples, respectively. No significant differences were detected between HAD and FZ+HAD powders.

Regarding color, L* values showed that HAD powder was significantly darker than the FD sample, while FZ+HAD power was the darkest. The three powders' hue angle were also significantly different. FD powder presented the higher hue angle, followed by HAD and FZ+HAD samples.

4. Discussion

4.1 Macronutrients and fiber composition

The values of mango peels' total carbohydrates, fat, protein, and ash (section 3.1) agree with data reported previously (Garcia-Amezquita et al., 2018; Marçal & Pintado, 2021). Regarding the effect of drying, FD and HAD caused no changes in mango peels' macronutrients, which is corroborated by Garcia-Amezquita et al. (2018). On the other hand, FZ+HAD reduced total fat, which probably occurred during freezing since Marçal et al. (2024) reported a similar decrease in mango peels submitted to the same freezing conditions. Mango peels are not an important source of fat, so reducing this macronutrient has no relevant impact on their nutritional value. The three drying methods reduced the moisture content to less than 10 g/ 100 g FW, which is important for decreasing water activity and consequently extending mango peels' shelf life by reducing spoilage microorganisms and enzymes' activity (Dukare et al., 2022; Ma et al., 2023).

FD, HAD, and FZ+HAD powders were a great source of fiber since a dose of 100 g DW provided 1.33 times the Adequate Intake (25 g/ day) for this nutrient (EFSA, 2010). Mango peels' soluble and insoluble fiber vary depending on the cultivar and ripening stage (Ajila & Rao, 2013). For instance, Geerkens, Nagel, et al. (2015) studied peels from the same mango cultivar and found values very similar to those presented in this study, while Garcia-Amezquita et al. (2018) reported higher values in Ataulfo mango peels. Like the present work, Geerkens, Nagel, et al. (2015) and Garcia-Amezquita et al. (2018) detected no differences between the fiber content of FD and HAD mango peels.

The polymers that compose fibers vary depending on raw materials and affect their technological properties and health benefits (Elleuch et al., 2011). In the present study, all polymers that composed powders' fiber, excluding lignin, a phenolic polymer, were hydrolyzed in monomeric sugars (Sluiter et al., 2008). The analysis of these simple sugars

460 enabled the deduction of the polymers that composed powders' soluble and insoluble fiber
461 (Ajila & Rao, 2013; Elleuch et al., 2011; Sluiter et al., 2008). According to previous studies,
462 in insoluble fibers, glucose arose from cellulose, while galactose, xylose, and arabinose came
463 from hemicellulose (Ajila & Rao, 2013; Elleuch et al., 2011; Sluiter et al., 2008). Hence, the
464 results (section 3.2) suggested that the main polymer in the insoluble fiber of the three
465 powders was cellulose (> 30% of insoluble fiber), while the abundance of hemicellulose and
466 acid-insoluble lignin was similar (c.a. 16% of insoluble fiber). The acid insoluble lignin
467 content can be slightly overestimated due to the presence of acid insoluble resistant protein
468 since it was not quantified due to methodological constraints. In soluble fiber, the main
469 constituent was uronic acids (38 – 42% of soluble fiber). Uronic acids include galacturonic
470 acids, the main constituent of pectin (Elleuch et al., 2011). Furthermore, galactose and
471 arabinose can also come from branch units of pectin (Elleuch et al., 2011). Glucose was the
472 least abundant sugar in soluble fiber (3 – 6% of soluble fiber), and, possible came from
473 soluble glucans (Ajila & Rao, 2013). Umbreen et al. (2015) reported that cellulose and pectin
474 were the main polymers in mango peels' fiber, followed by hemicellulose and lignin, which
475 corroborated the results presented in this study.

476 To our knowledge, no previous studies evaluated the impact of FD and HAD on other
477 polymers from mango peels' fiber besides pectin. Geerkens, Nagel, et al. (2015) reported that
478 the yield of alcohol-insoluble solids (composed mainly of pectin) was higher in FD mango
479 peels than in HAD ones. The methodologies used to quantify pectin in the present study and
480 Geerkens, Nagel, et al. (2015) were different, which can explain the discrepant findings. FD,
481 HAD, and FZ+HAD powders' insoluble and soluble fiber differed in glucose content, which
482 suggested that these powders had different cellulose and glucan amounts, respectively. These
483 differences can result directly from disrupting glucans and cellulose during processing or
484 disrupting other polymers that compose fiber, leading to cellulose and glucans concentration

(Garau et al., 2007). Cellulose is among the most resistant polymers that compose dietary fibers (Garau et al., 2007). More studies are needed to clarify it.

Overall, the three drying methods were adequate for fiber preservation. Fiber recovery, namely pectin extraction, represents one of the more usual and economically valuable applications of mango peels in the food industry (Gupta et al., 2022; Marçal & Pintado, 2021).

4.2 Antioxidants and antioxidant activity

The major antioxidants in mango peels are vitamin C, phenolic compounds, and carotenoids (Marçal & Pintado, 2021). This section discusses the effects of FD, HAD, and FZ+HAD on these antioxidants and antioxidant activity.

4.2.1 Vitamin C

FS, FD, HAD, and FZ+HAD samples were a great source of vitamin C since a dose of 100 g DW provided 5.42, 4.92, 2.46, and 1.50 times, respectively, the Average Requirements (90 mg/ day) (EFSA, 2017). The vitamin C values found in this study were within the range reported by Marçal & Pintado (2021). Moreover, Troiani et al. (2022) reported similar values in fresh mango peels from Palmer, Keith, Haden, and Espada Vermelha cultivars (143.47 - 430.31 mg/ 100 g DW). On the other hand, Sogi et al. (2013) found lower vitamin C values (FD: 75.48 mg/ 100 g DW; HAD: 84.74 mg/ 100 g DW) in Tommy Atkins mango peels. Environmental factors, namely light exposure, highly affect vitamin C synthesis (Paciolla et al., 2019). So, possible differences in cultivation conditions can explain these discrepant values.

To our knowledge, this was the first study to simultaneously assess the vitamin C content in FS and FD mango peels. Although FD showed the best performance regarding vitamin C retention, the reduction caused by this drying method was significant (section 3.1).

Several previous studies also reported significant reductions in vitamin C of FD fruits (e.g., mango and pitaya: -28%, papaya -26%, apple: -3%) (Ma et al., 2023). Regarding the impact of HAD, Roa-Tort et al. (2024) reported a reduction of 8.14% of vitamin C in HAD (45 ºC, 24 h) mango peels compared with fresh ones, while Sogi et al. (2013) found no differences between vitamin C content of HAD (60 ºC, 4 h) and FD mango peels. In the present study, HAD had more harmful effects on vitamin C content (section 3.1). Differences in temperatures and drying times used can explain these discrepant findings. Dukare et al. (2022) observed that mango peels HAD at 50 ºC had a higher vitamin C content than those dried at 70 and 80 ºC. Like FZ+HAD, drum drying (130 – 146 ºC; 14 – 28 s) did not seem to be an adequate alternative method to preserve vitamin C since Troiani et al. (2022) and Antoniolli et al. (2023) reported vitamin C reductions between 61% and 86% in drum-dried mango peels.

4.2.2 Phenolic compounds and antioxidant activity

The profiles of mango peels' free and bound phenolic compounds found in this study agree with data previously reported (Ancos et al., 2018; Gupta et al., 2022; Marçal & Pintado, 2021; Pacheco-Ordaz et al., 2018). For example, Pacheco-Ordaz et al. (2018) and Ancos et al. (2018) also identified mangiferin as the main free phenolic compound. Furthermore, Pacheco-Ordaz et al. (2018) also reported a higher diversity of compounds in BBPC extracts than in ABPC extracts and identified gallic acid as the main phenolic compound in both extracts.

The effect of HAD and FD on mango peels' total free phenolic compounds has been widely studied (de Brito Araújo et al., 2021; Dukare et al., 2022; Roa-Tort et al., 2024; Santos et al., 2023; Sogi et al., 2013). These previous works corroborate this study (section 3.3). For instance, Sogi et al. (2013) reported that the total phenolics content in HAD samples was 27% lower than in FD samples; Dorta et al. (2012) observed a reduction of 53.06% in

mango peels submitted to HAD preceded by freezing compared with FD; and Santos et al. (2023) found significant reductions in HAD samples (-44.57% and -57.93% in samples dried at 70 ºC and 60 ºC, respectively) compared with fresh ones.

To our knowledge, no previous studies simultaneously compared the free phenolics profile of FS, FD, and HAD mango peels. Nevertheless, Ancos et al. (2018) evaluated this parameter in FD and HAD mango peels, while Berardini et al. (2005) assessed it in FD and HAD mango peels previously frozen at -20 ºC. As expected and in agreement with the present study (section 3.3), Ancos et al. (2018) and Berardini et al. (2005) also observed that the amount of some free phenolic compounds varied significantly depending on the drying method. Succinctly, Ancos et al. (2018) found that FD mango peels had a higher content of flavonoids and xanthones, while HAD samples had a higher amount of gallic acid and some other gallates, which corroborate the present study (section 3.3). Regarding Berardini et al. (2005), they also reported reductions in mangiferin and some quercetin 3-O-glycosides. However, the magnitude of reductions reported by Berardini et al. (2005) was lower than in the present study, which is probably justified by distinct drying times.

The increases of free catechin and gallic acid in FD and HAD samples (section 3.3), respectively, can be related to two distinct phenomena, the conversion of some bound phenolics into free ones due to changes in mango peels' microstructure, as discussed below, and the splitting of complex phenolics into simple ones (Ancos et al., 2018; Lang et al., 2019; Ma et al., 2023).

To our knowledge, this was the first work that evaluated the effect of drying on mango peels' bound phenolic compounds. According to previous studies, two different phenomena with opposite effects on bound phenolic compounds can occur during drying (Lang et al., 2019; Ma et al., 2023). On the one hand, water loss during drying can damage the foods' microstructure, favoring the release of matrix-bound phenolics and consequently decreasing

559 the amount of bound phenolic compounds (Lang et al., 2019; Ma et al., 2023). In drying
560 methods preceded by freezing, like FD and FZ+HAD, the release of bound phenolic
561 compounds can be heightened since freezing also damages foods' microstructure (Li et al.,
562 2020; Ma et al., 2023; Marçal et al., 2024). On the other hand, the drying conditions can
563 favor the complexation of some initial free phenolic compounds with structural constituents
564 increasing bound phenolic compounds (Lang et al., 2019). This last phenomenon is a possible
565 explanation for the increase of 3,4-dihydroxybenzoic acid and ferulic acid in BBPC extract
566 from the HAD sample, while the first one justified the drastic reduction of total and some
567 individual bound phenolic compounds, observed in all dried samples compared with FS
568 mango peels.

569 Additionally, the milling process performed after drying can also contribute to changes
570 in mango peels' microstructure and conditioning the bound phenolic compounds content.
571 Although before extraction of phenolic compounds, all samples were submitted to the same
572 milling process, it resulted in powders with different particle sizes distribution (section 3.6).
573 The particle sizes of powders also influence the content of free and bound phenolic
574 compounds (Guo et al., 2012). Hence, considering that HAD and FZ+HAD had a similar
575 particle size distribution and the data presented in section 3.3, it can be concluded that HAD
576 had a better performing preserving bound phenolics compounds than FZ+HAD and,
577 consequently, is a better choice when the desired final product is antioxidant fiber. Regarding
578 FD, more studies are needed to clarify its performance compared with HAD and FZ+HAD
579 since the particles size distribution can have acted as a confounding variable.

580 The present study showed that mango peels had a high antioxidant activity (section 3.5)
581 which agrees with previous literature (Dorta et al., 2012; Gupta et al., 2022; Marçal &
582 Pintado, 2021; Sogi et al., 2013). As expected, the drying methods that have harmful effects
583 on phenolic compounds also impaired mango peels' antioxidant activity (Dorta et al., 2012).

584 *4.2.3 Carotenoids*

585 Like the present study, Ruales et al. (2018) also determined the carotenoids profile of
586 Tommy Atkins mango peels and found the same three carotenoids (β-carotene, lutein, and
587 violaxanthin). However, in Ruales et al. (2018), the most abundant carotenoid was lutein
588 instead of β-carotene. In its turn, Ancos et al. (2018) corroborated the present study since
589 they identified β-carotene as the main carotenoid in FD and HAD peels from Ataulfo
590 mangoes. Besides the cultivation conditions, other factors like mango cultivar (namely
591 different colored cultivars), postharvest processing, ripening stage, and different epicarp and
592 mesocarp proportions contribute to mango peels' chemical variability and can explain these
593 discrepancies (Marçal et al., 2024; Nagel et al., 2017; Ranganath et al., 2018).

594 Several previous studies evaluated the impact of FD and HAD on mango peels' total
595 carotenoids and β-carotene (Ancos et al., 2018; de Brito Araújo et al., 2021; del Pilar
596 Sanchez-Camargo et al., 2019; Roa-Tort et al., 2024; Sogi et al., 2013). However, to our best
597 knowledge, the present study was the first one to assess the effect of these drying methods on
598 mango peels' violaxanthin, which is a potent peroxidation inhibitor (50 times stronger than β-
599 carotene) (Takemura et al., 2021). The high susceptibility of violaxanthin to drying, including
600 FD, observed in the present study (section 3.4) was also reported in other raw materials by
601 previous works (Topuz et al., 2011). For instance, FD and HAD reduced the violaxanthin by
602 45.56% and 52.96% in paprika, respectively (Topuz et al., 2011). Marçal et al. (2024)
603 showed that mango peels' violaxanthin was also susceptible to freezing (-51%).

604 The authors found no previous studies that compared the lutein content of FS and FD or
605 HAD mango peels. However, Ancos et al. (2018) compared FD and HAD mango peels and
606 observed that both had the same lutein content. Furthermore, Ancos et al. (2018) did not
607 detect zeaxanthin in the FD sample but found this carotenoid in HAD mango peels. The
608 HPLC method used in the present study to quantify carotenoids did not enable the separation

609 of lutein and zeaxanthin since they had the same retention time. So, the values of lutein
610 presented in Section 3.4 can correspond to the sum of the two compounds, and the increase
611 observed in HAD samples can have resulted from the rise of lutein, zeaxanthin, or both.
612 Considering the results reported by Ancos et al. (2018), it can be speculated that this increase
613 occurred due to zeaxanthin rise. More studies are needed to clarify the mechanisms that lead
614 to this increase.

615 Regarding β-carotene, the results of the present study agree with Ancos et al. (2018)
616 since they found no differences between β-carotene content of FD and HAD mango peels. On
617 the other hand, del Pilar Sanchez-Camargo et al. (2019) reported a higher β-carotene content
618 in FD than in HAD mango peels. These discrepant results can be related to differences in
619 mango peels' microstructure (Ancos et al., 2018). For instance, Ancos et al. (2018) reported
620 better retention of β-carotene in HAD mango peels than in HAD mango pasta and attributed
621 these discrepant results to differences in the matrix of pasta and peels.

622 Overall, this study showed that FD and HAD are adequate for preserving mango peels'
623 carotenoids, while FZ+HAD significantly reduces them, so it is inappropriate.

624 *4.2.3 Different drying mechanisms and antioxidants preservation*

625 As expected, FD was better for preserving most of the antioxidants than HAD and
626 FZ+HAD. Different drying mechanisms justify the distinct performances. In HAD and
627 FZ+HAD, the water was removed by evaporation due to contact of mango peels with hot air
628 (Geerkens, Miller-Rostek, et al., 2015). On the other hand, in FD, the water was removed by
629 sublimation under freezing temperatures and low-pressure conditions, which minimized the
630 loss of thermolabile compounds like vitamin C, flavonoids, and xanthones (Geerkens, Miller-
631 Rostek, et al., 2015). Furthermore, the exposure of samples to oxygen was lower in FD than
632 in HAD (due to low-pressure conditions versus forced air circulation), which also decreased

633 the deterioration of antioxidant compounds caused by reactions with oxygen (Geerkens,
634 Miller-Rostek, et al., 2015). Additionally, during HAD and FZ+HAD, depending on
635 temperatures, enzymes like polyphenol oxidase can remain active until the water activity is
636 lower than 0.6 (Geerkens, Miller-Rostek, et al., 2015).

637 FZ+HAD performed worse preserving antioxidants than HAD. Marçal et al. (2024)
638 submitted mango peels to the same freezing conditions as in the present work, and no
639 significant reductions were detected in vitamin C. Moreover, all individual free phenolic
640 compounds were preserved or increased after freezing, while in carotenoids, only
641 violaxanthin suffered a significant reduction (Marçal et al., 2024). These previous results
642 indicated that most of the reductions of antioxidants in FZ+HAD samples occurred during the
643 drying step. Marçal et al. (2024) observed that freezing damaged mango peels'
644 microstructure since the porous became larger or ruptured. These changes in frozen mango
645 peels' microstructure can justify the bad performance of FZ+HAD regarding the retention of
646 antioxidants since the food structure plays an important role in protecting bioactive
647 compounds from oxidation during drying (Ancos et al., 2018). In other words, the changes
648 caused by freezing in mango peels' microstructure make phenolic compounds more
649 extractable but also more susceptible to adverse conditions (e.g., temperature and oxygen)
650 applied during HAD (Ancos et al., 2018; Marçal et al., 2024).

651 Although FD is the golden standard for preserving antioxidant compounds, it is an
652 environmentally and economically expensive method since a high amount of energy needs to
653 be spent to maintain sublimation conditions (Ancos et al., 2018; del Pilar Sanchez-Camargo
654 et al., 2019; Garcia-Amezquita et al., 2018). Moreover, it is a time-consuming method, and
655 the equipment is expensive (Ancos et al., 2018; del Pilar Sanchez-Camargo et al., 2019). On
656 the other hand, HAD is a simple, cheap, and fast method. So, even when a mango peel
657 powder with antioxidant properties is the desired final product, HAD could be the most

658 economically and environmentally sustainable method for preserving mango peels. Hence,
659 developing new and sustainable strategies for minimizing losses of mango peels' antioxidants
660 during HAD is an opportunity for future research. Previous studies with other raw materials
661 showed that pretreatment with natural extracts can be a viable strategy for improving the
662 quality of HAD products (Ahmad et al., 2024). However, to our best knowledge, this has not
663 yet been studied in mango peels.

664 **4.3 Technological properties**

665 Geerkens, Nagel, et al. (2015) observed that applying the same milling procedure to FD
666 and HAD alcohol insoluble solids from mango peels, the first ones resulted in powders with a
667 lower mean particle size, which corroborates the present study. The differences in particle
668 size distribution of FD and HAD or FZ+HAD mango peels possibly impacted other mango
669 peel powders' characteristics, namely bound phenolics, and water and oil absorption
670 capacities (Elleuch et al., 2011; Guo et al., 2012).

671 The water and oil absorption capacities reported in the present study for FD and HAD
672 mango peel powders were similar to those found by Sogi et al. (2013). Furthermore, Sogi et
673 al. (2013) also observed that FD powders had a higher capacity to absorb oil, while HAD
674 powder showed a better performance regarding water absorption capacity. Powders' water
675 and oil absorption capacities are relevant for their application in food products. For instance,
676 powders with high oil absorption capacity contribute to stabilize high-fat products, while high
677 water absorption capacity avoids synaeresis and increases laxative properties (Elleuch et al.,
678 2011).

679 The darkening of mango peels during drying occurred due to enzymatic browning and
680 Maillard reactions. Moreover, the decrease of hue angle from can be related with chlorophyll
681 degradation (Dorta et al., 2012; Dukare et al., 2022). The results (table 7 and section 3.6)
682 suggested that all these deterioration reactions were more intense in FZ+HAD followed by

HAD, and FD. Like in antioxidants preservation (section 4.2.2), different drying mechanisms and changes in cells microstructure due to freezing (in FZ+HAD samples) explain these differences.

5. Conclusion

Although the impact of drying on mango peels' composition and properties has been widely studied during the last decades, this work brought new knowledge about important fields that have not been explored until now. To our knowledge, this study was the first to: compare the HAD of fresh and frozen mango peels; evaluate the impact of FD, HAD, FZ+HAD, or any drying method on mango peels' bound phenolic compounds and violaxanthin; assess the vitamin C content in fresh and FD mango peels simultaneously, and therefore clarify the effect of FD on this micronutrient; and evaluate simultaneously free phenolics profile and lutein content of FS, FD, and HAD mango peels. Moreover, the authors found no previous studies about the impact of FD and HAD on other polymers from mango peels' fiber besides the pectin.

Fresh mango peels were a better source of vitamin C, violaxanthin, and bound phenolic compounds than all powders studied. Overall, FZ+HAD performed worse, preserving all antioxidants (vitamin C, phenolic compounds, and carotenoids) than HAD and FD. However, like the other drying methods, FZ+HAD caused no relevant changes in mango peels' macronutrients as well as in soluble fiber's uronic acids content. Furthermore, the water and oil capacities were similar in HAD and FZ+HAD powders. So, FZ+HAD can be an adequate method for digestible carbohydrates and fiber recovery or, more specifically, pectin extraction. Nevertheless, FZ+HAD is inefficient when antioxidant fiber is the desired final product due to its harmful effect on bound phenolic compounds.

As expected, overall, FD preserved vitamin C, free phenolic compounds, and violaxanthin better than HAD. Nevertheless, HAD powder had a similar β-carotene content

and higher amount of lutein and bound phenolic compounds than FD samples. However, more studies are needed to clarify the FD performance regarding bound phenolics preservation compared with HAD and FZ+HAD, since different particle size distributions can influence other variables. Developing new and sustainable strategies for minimizing losses of mango peels' antioxidants during HAD (e.g., pretreatment with natural extracts) is an opportunity for future research.

Declarations of interest: none

Author contributions

Sara Marçal: methodology; formal analysis; investigation; data curation; writing - original draft; visualization; **Ana A. Vilas-Boas:** methodology; **Débora A. Campos**: methodology; validation; writing - review & editing; supervision **Manuela Pintado**: conceptualization; methodology; validation; resources; writing - review & editing; supervision; project administration; funding acquisition.

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974 **Tables**

Table 1. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on mango peels' nutritional composition.

	Dry Weight g/ 100 g FW	Total Carbohydrates g/ 100 g DW	Total fiber g/ 100 g DW	Soluble fiber g/ 100 g DW	Insoluble fiber g/ 100 g DW	Protein g/ 100 g DW	Fat g/ 100 g DW	Ash g/ 100 g DW	Vitamin C mg/ 100 g DW
FS	16.83±0.68^{B}	90.15±0.54^{B}	33.62±1.43^{A*}	16.57±0.60^{A*}	17.69±0.33^{A*}	5.88±0.74^{A}	1.92±0.05^{A*}	2.05±0.34^{B}	488.22±20.49^{A}
FD	94.37±0.46^{A}	90.63±0.36^{AB}	33.31±0.96^{A}	15.57±0.76^{A}	17.44±1.10^{A}	4.96±0.33^{A}	1.89±0.04^{A}	2.53±0.00^{AB}	442.49±7.57^{B}
HAD	90.83±3.27^{A}	90.24±0.24^{B}	33.62±1.43^{A*}	16.57±0.60^{A*}	17.69±0.33^{A*}	4.95±0.08^{A}	1.92±0.05^{A*}	2.89±0.20^{A}	221.62±2.62^{C}
FZ+HAD	93.47±0.78^{A}	91.26±0.26^{A}	33.09±1.10^{A}	16.41±1.06^{A}	17.69±0.11^{A}	4.81±0.22^{A}	1.56±0.02^{B}	2.36±0.03^{B}	134.74±1.90^{D}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). *The soluble, insoluble, and total fiber contents and fat amount in fresh mango peels were estimated using the HAD sample since the methodologies applied to quantify these nutrients are inadequate for fresh samples. FW – fresh weight; DW – dry weight; FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 2. Composition of soluble and insoluble fiber from freeze dried, hot air dried, and frozen and hot air-dried mango peels.

		Acid insoluble lignin and protein g/ 100 g of fiber	Uronic Acids g/ 100 g of fiber	Glucose g/ 100 of fiber	Galactose g/ 100 g of fiber	Xylose g/ 100 g of fiber	Arabinose g/ 100 g of fiber
Soluble Fiber	**FD**	-	39.87±1.97^{A}	4.31±0.08^{B}	21.08±0.82^{A}	nd	17.58±1.18^{A}
	HAD	-	42.55±1.34^{A}	3.31±0.05^{C}	21.11±0.29^{A}	nd	16.86±0.77^{A}
	FZ+HAD	-	38.22±1.92^{A}	6.31±0.18^{A}	20.38±0.78^{A}	nd	17.47±0.30^{A}
Insoluble fiber	**FD**	16.73±1.17^{A}	10.70±0.44^{C}	33.50±0.38^{C}	8.09±1.33^{A}	5.83±0.07^{A}	2.24±0.26^{A}
	HAD	15.72±0.75^{A}	14.15±0.31^{A}	36.34±0.41^{B}	7.30±0.82^{A}	5.88±0.12^{A}	2.17±0.54^{A}
	FZ+HAD	15.97±0.76^{A}	12.96±0.57^{B}	39.03±0.01^{A}	6.95±0.49^{A}	6.34±0.39^{A}	2.44±0.82^{A}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h); nd – not detected.

Table 3. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on free and bound phenolic compounds released from mango peels through methanolic extraction, basic and acid hydrolyses, respectively.

	Methanolic extraction µg / g DW				Basic hydrolysis µg / g DW				Acid hydrolysis µg / g DW			
	FS	**FD**	**HAD**	**FZ+HAD**	**FS**	**FD**	**HAD**	**FZ+HAD**	**FS**	**FD**	**HAD**	**FZ+HAD**
Total phenolic compounds	12383.18±2147.47^{A}	10624.73±1392.74^{A,B}	7680.63±103.33^{B,C}	6313.85±333.03^{C}	1075.56±270.66^{A}	314.40±41.88^{B}	631.06±46.08^{B}	644.95±17.84^{B}	221.95±11.47^{A}	68.73±11.01^{C}	113.56±12.62^{B}	120.91±20.60^{B}
Mangiferin	963.77±116.65^{A}	863.50±10.66^{A}	632.50±10.11^{B}	443.81±3.26^{C}	34.35±5.84^{A}	9.51±2.87^{B}	28.84±6.85^{A}	22.87±6.61^{A}	9.21±1.33^{A}	2.18±0.52^{B}	6.92±1.50^{A}	6.25±1.45^{A}
Gallic acid	610.68±52.39^{B}	650.32±20.53^{B}	830.29±9.07^{A}	508.87±8.96^{C}	533.04±101.22^{A}	42.65±1.63^{C}	152.66±26.58^{BC}	207.74±7.32^{B}	92.81±25.13^{A}	12.27±0.27^{B}	23.25±0.03^{B}	28.26±1.21^{B}
Penta-O-galloyl-β-D-glucose	401.17±50.00^{A}	384.21±9.76^{A}	358.72±13.26^{A}	160.83±50.81^{B}	nd	nd	nd	nd	nd	nd	nd	nd
Methyl gallate	323.74±53.86^{A}	299.31±2.22^{A}	188.72±13.61^{B}	93.38±5.49^{C}	nd	nd	nd	nd	nd	nd	nd	nd
Catechin	409.16±19.86^{B}	485.27±6.62^{A}	249.03±15.98^{C}	118.00±14.15^{D}	nd	nd	nd	nd	nd	nd	nd	nd
Quercetin-3-O-galactoside[a]	490.95±65.86^{A}	464.20±3.42^{A,B}	381.88±0.79^{B}	513.04±31.56^{A}	19.99±1.74^{A}	8.72±1.64^{C}	18.71±1.72^{AB}	14.54±3.24^{B}	nd	nd	nd	nd
Quercetin 3-β-D-glucoside	183.09±16.69^{A}	185.26±0.85^{A}	144.87±0.77^{B}	138.93±7.56^{B}	20.53±4.39^{A}	7.97±1.80^{B}	20.38±2.05^{A}	15.76±0.39^{A}	nd	nd	nd	nd
Quercetin 3-O-α-L-arabinopyranoside	79.17±11.46^{A}	80.08±0.74^{A}	63.84±0.96^{B}	53.08±2.02^{B}	7.56±1.54^{A}	3.54±0.01^{B}	7.71±0.69^{A}	4.65±1.44^{B}	nd	nd	nd	nd
Quercetin-3-O-α-L-arabinofuranoside	52.05±7.71^{A}	50.59±0.16^{A}	39.70±0.79^{B}	35.49±1.54^{B}	4.89±1.00^{A}	2.29±0.36^{B}	4.71±0.32^{A}	3.18±0.92^{AB}	nd	nd	nd	nd
Ferulic acid	nd	nd	nd	nd	6.26±1.32^{B}	3.45±0.37^{C}	8.81±0.65^{A}	6.48±0.08^{B}	nd	nd	nd	nd
p-Coumaric acid	nd	nd	nd	nd	13.29±0.04^{A}	10.18±0.24^{B}	14.12±1.27^{A}	11.24±0.13^{B}	2.01±0.21^{A}	1.38±0.15^{B}	2.04±0.21^{A}	1.27±0.09^{B}
3,4-dihydroxybenzoic acid	nd	nd	nd	nd	47.59±3.43^{B}	52.72±8.84^{B}	81.82±14.95^{A}	23.23±0.02^{C}	nd	nd	nd	nd
4-hydroxybenzoic acid	nd	nd	nd	nd	66.26±9.69^{A}	16.69±1.20^{C}	50.39±7.23^{A,B}	46.15±10.79^{B}	25.86±1.48^{A}	2.58±0.25^{C}	6.12±0.43^{B}	5.63±0.87^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same row indicate statistically significant differences between samples ($p < 0.05$). [a]In HPLC method used quercetin-3-O-galactoside and ellagic acid had the same retention time, so these values can correspond to these both compounds. DW – dry weight; nd –

not detected; FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 4. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on mango peels' carotenoids.

	β-carotene µg/ g DW	Lutein[a] µg/ g DW	Violaxanthin µg/ g DW
FS	388.97±11.56^{A}	15.12±1.14^{B}	8.47±0.11^{A}
FD	396.77±34.59^{A}	16.16±1.60^{AB}	4.28±0.84^{B}
HAD	429.25±20.38^{A}	18.96±1.46^{A}	1.11±0.09^{C}
FZ+HAD	249.91±32.64^{B}	11.76±1.35^{C}	nd

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). [a]In the HPLC method lutein and zeaxanthin had the same retention time, so these values can correspond to these both compounds. DW – dry weight; nd – not detected; FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 5. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on antioxidant activity of free and bound phenolic compounds released from mango peels through methanolic extraction and basic and acid hydrolyses, respectively.

		ABTS	DPPH
		μg of Trolox eq. / g DW	μg of Trolox eq. / g DW
Free phenolic compounds	**FS**	30567.99±1940.59^{A}	28429.99±3251.63^{A}
	FD	27262.52±1865.66^{A}	24766.32±1729.97^{A}
	HAD	17384.16±697.05^{B}	14717.43±1352.84^{B}
	FZ+HAD	16804.20±2292.16^{B}	13706.52±2313.19^{B}
Bound phenolic compounds (basic hydrolysis)	**FS**	3259.78±809.41^{A}	2591.03±757.94^{A}
	FD	621.56±28.51^{B}	390.29±32.31^{C}
	HAD	1364.10±117.82^{B}	1153.44±109.63^{BC}
	FZ+HAD	1622.91±57.97^{B}	1380.51±31.00^{B}
Bound phenolic compounds (acid hydrolysis)	**FS**	510.02±92.15^{A}	346.81±51.87^{A}
	FD	128.20±14.41^{B}	72.33±14.02^{C}
	HAD	217.18±37.06^{B}	179.54±8.71^{B}
	FZ+HAD	258.42±55.16^{B}	214.32±55.81^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). FS – fresh sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 6. Particle size distribution in freeze dried, hot air-dried, and frozen and hot air-dried samples.

	>500 µm g/ 100 g	250 – 500 µm g/ 100 g	150 – 250 µm g/ 100 g	100 – 150 µm g/ 100 g	50 – 100 µm g/ 100 g	<50 µm g/ 100 g
FD	6.03±0.29^{C}	11.79±0.16^{C}	13.92±0.97^{B}	12.62±1.20^{A}	31.15±4.95^{A}	24.49±4.33^{A}
HAD	18.26±1.28^{A}	26.88±0.96^{B}	17.22±1.06^{A}	10.90±0.45^{A}	15.70±1.58^{B}	11.03±1.61^{B}
FZ+HAD	14.75±0.41^{B}	29.16±0.67^{A}	18.71±0.07^{A}	12.48±0.48^{A}	15.46±0.88^{B}	9.45±1.31^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). DW – dry weight; W – washed sample; FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).

Table 7. Impact of freeze drying, hot air drying, and hot air drying preceded by freezing on mango peel powders' color, water absorption capacity, and oil absorption capacity.

	Color		Water Absorption Capacity	Oil Absorption Capacity
	L*	Hue Angle	g of water/ g of sample	g of oil/ g of sample
FD	20.21±0.46^{A}	89.54±0.01^{A}	5.04±0.07^{B}	2.03±0.11^{A}
HAD	15.62±0.23^{B}	81.42±0.20^{B}	6.29±0.25^{A}	1.40±0.07^{B}
FZ+HAD	14.39±0.41^{C}	77.65±1.15^{C}	6.23±0.37^{A}	1.28±0.15^{B}

Each value was expressed as mean ± standard deviation (n = 3). Different letters in the same column indicate statistically significant differences between samples ($p < 0.05$). FD – freeze dried sample; HAD - hot air-dried sample (at 65 ºC for 48 h); FZ+HAD – samples submitted to freezing (at -20 ºC for 30 days) followed by hot air drying (at 65 ºC for 48 h).