








VOLATILE AND CARBOHIDRATE PROFILE OF PERSIMMON EXTRACTS

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Abstract

Persimmon (*Diospyros kaki*) is an orange-coloured fruit renowned for its sweet, honey-like flavour when ripe. It is particularly valued for its versatility in culinary applications and as a rich source of essential nutrients, including vitamin C, pro-vitamin A, dietary fibre, and minerals. Despite these benefits, persimmon faces significant challenges due to its highly perishable nature, which limits its shelf life and utilization potential. One promising solution is the incorporation of persimmon derivatives into stable food products to reduce waste and enhance the value chain of this nutritious fruit. In this study, we aimed to examine the potential of persimmon for the development of confectionery food products by producing persimmon powder and syrup and analysing their chemical profiles. Volatile aroma profiles were evaluated using solid-phase microextraction (SPME) coupled with gas chromatography–mass spectrometry (GC–MS), while carbohydrate profiles were assessed using thin-layer chromatography (TLC) and ion chromatography (IC). Among the volatiles identified, furfural, a compound with a characteristic almond-like odour and significant application as a food additive, emerged as the most abundant volatile in both persimmon derivatives. Sugar profiles revealed that main sugars are sucrose, glucose, and fructose, with little to no fructooligosaccharides detected. These findings support the potential for persimmon derivatives to be used as ingredients in confectionery product formulations.

Keywords: food insecurity, carbohydrates, persimmon, food supply, volatiles.

Introduction

Persimmon (*Diospyros kaki*) is an orange-coloured fruit renowned for its sweet, honey-like flavour when ripe, which is highly valued for its versatility in culinary applications. Although it is originated in East Asia - particularly China, Japan, and Korea - its use has extended throughout the globe (Gea-Botella et al., 2021). It is rich in natural carbohydrates and is commonly processed into juice, jam, and other sweet products. Globally, persimmons are recognized for their dual role as a nutritional and medicinal resource. The fruit contains proteins, sugars, vitamins (A, B6, B12, C, D, E), carotenoids, polyphenols, flavonoids, and essential minerals such as potassium, calcium, and iron (Manach et al., 2004; Butt et al., 2015). Historically, persimmon leaves and fruits have been utilized in the treatment of various conditions such as stroke, apoplexy, angina, internal haemorrhage, paralysis, frostbite, and burns (Xie et al., 2015). More recent studies have highlighted the pulp, skin, and leaves of persimmons for their potential antioxidant, anti-edema, anti-hyperlipidaemic, and anti-diabetic effects (Xie et al., 2015; Kim et al., 2018). The high content of flavonoids and their antioxidant properties, which neutralize reactive oxygen species, play a significant role in persimmon's traditional use for conditions like hypertension, atherosclerosis, and metabolic disorders. These phytochemicals are also responsible for its documented anti-diabetic and anti-hyperlipidaemic effects (Manach et al., 2004; Xie et al., 2015).

Despite their promising culinary uses and health benefits, several tons of persimmons are discarded globally because of the rather low shelf life of persimmon fruits plus different factors including strict

regulations by governments, high quality standards of supermarkets, and high expectations of consumers regarding fruit colour, size and shape (Porter et al., 2018; Gea-Botella et al., 2021). In Uzbekistan, thousands of tons of persimmons are wasted every year because of poor storage, late harvest, or inadequate market demand (Normakhmatov et al., 2024).

Even though persimmon is used in confectionery products and advances have been made in dried persimmon production, the fruit's processing remains relatively limited, and its large-scale industrial utilization has lagged behind that of other commercially significant fruits (Tsurunaga et al., 2021; Normakhmatov et al., 2024). Although some countries, including Uzbekistan, export dried raisins of the fruit, a more expansive range of products such as jams, vinegars, and ice creams, could potentially bolster the fruit's market. The incorporation of persimmon into those confectionery products could help to increase its shelf life and reduce fruit waste. A deeper investigation into both primary and by-product components of persimmon processing is therefore essential to help reduce these losses, extend shelf life, and explore new uses (Butt et al., 2015; Normakhmatov et al., 2024; H Al-Qaisi et al., 2025). Although persimmons have been grown in Uzbekistan since the last century, studies on local varieties of either fruits or derivatives are scarce.

This study aimed to develop and characterize a persimmon-based powder and syrup, obtained from a local variety of the fruit, for future confectionery applications, focusing on their volatile and sugar profiles to determine suitability for use in future confectionery formulations. To evaluate flavour and

aroma compounds in the persimmon extracts, volatile profiles were analysed using solid-phase microextraction (SPME) and gas chromatography–mass spectrometry (GC–MS). Besides, carbohydrate profiles were qualitatively investigated by thin layer chromatography (TLC) and ion chromatography (IC).

Materials and Methods

Raw Materials

For this study we used ripe Zenji-maru persimmon fruits grown in the Tashkent and Kashkadarya regions, sourced from local farmers. Colour of fruits ranged from flame to deep red. We based our variety selection on a previous study where persimmon varieties grown in Uzbekistan were evaluated for their suitability for drying (Normakmatov et al., 2024).

Extraction of syrup and powder from persimmon fruits
Persimmon fruits were washed in cold water and dried. To obtain persimmon extracts, the fruits were cut into pieces, and the flesh and seeds were separated, thus obtaining the ‘pulp’. The pulp was placed in a blender with water added at a ratio of 3:1 and pureed. The puree was then transferred to a container, water was added at a ratio of 3:2, and the mixture was boiled at 80 °C for 30 minutes. The resulting product was separated into phases in a Hettich (Tuttlingen, Germany) centrifuge, model EBA 200, at 3000 rpm. The separated aqueous phase was transferred to a separate container, and the remaining solid material underwent re-extraction twice following the same procedure, thus obtaining the ‘first extract’. Syrup was obtained by concentrating the first extract in a rotary evaporator apparatus IKA RV8 with a heating device IKA HB digital (IKA, China) equipped with a recirculating water chiller YHLT-10/30 (Green Distill, China) and a diaphragm vacuum pump KNF N 035.3 AN.18 (KNF, Germany) (13 mbars abs. ultimate vacuum, according to specification sheet) until the extract volume decreased by 50%. Powder was obtained by drying the pulp in a Spray Dryer (Wenming, China), model WM-SP1500S, at a temperature of 85–100 °C until powder was achieved. Dried material was then ground into powder using a mill. Weight was measured in a Kern (Balingen, Germany) analytical balance, model ABJ-4NM.

Volatile analyses

Analytical procedure was based on the method outlined by (Calvo-Gómez et al., 2004) with modifications. Samples were analysed by triplicate.

SPME Procedure

Divinylbenzene/ Carboxen/ Polydimethylsiloxane (DVB/CAR/PDMS) 50/30 µm SPME fibres and holders were sourced from Sigma-Aldrich (USA). Samples (1 g of persimmon powder -coded as P1- or persimmon syrup -coded as P3-) were loaded into 4 mL vials and hermetically sealed with septum-lined caps. Equilibration occurred at 30 °C for 1 h under static conditions. The SPME fibre assembly was introduced through the septum, secured in place, and exposed to the vial

headspace for 30 min at 30 °C. Following adsorption, the fibre was retracted and promptly transferred to the GC injection port for thermal desorption.

GC-MS Analyses

Volatile compounds were analysed using an Agilent 7890/5977 GC-MS (Agilent, USA) system equipped with an HP-INNOWax capillary column (30 m × 0.25 mm ID, 0.15 µm film thickness; max temperature: 270 °C) (Agilent, USA). Operational parameters included: Injection: 1 minute desorption at 250 °C in splitless mode. Carrier Gas: Helium at 3 mL/min constant flow. Oven Program: Initial temperature 40 °C (5 min hold), ramped at 20 °C/min to 240 °C (5 min final hold). MS Settings: Electron ionization (70 eV), ion source (230 °C), quadrupole (150 °C), full-scan acquisition (m/z 50–550). Compound identification utilized spectral matching against the NIST/EPA/NIH mass spectral library.

Carbohydrate analyses

For retention time comparison, a standard was prepared: Glucose (G), fructose (F), and sucrose (S) were acquired from Sigma- Aldrich (Sigma-Aldrich, USA). And fructooligosaccharides (FOS): 1-kestose (1-kestotriose; 1K), 1-nystose (1, 1-kestotetraose; 1N), and DP5 (1-Fructofuranosyl-D-Nystose) were from Wako Pure Chemical Industries (Osaka, Japan). Compounds were diluted to a concentration of 0.5 mg mL⁻¹ with deionized water (resistivity of 17 M Ω).

Thin Layer Chromatography

Analytical procedure was based on the method outlined in (Mellado-Mojica et al., 2022; Mellado-Mojica & López, 2015). The samples were diluted with deionized water (resistivity of 17 MΩ) to a concentration of 10 mg mL⁻¹ and then filtered through 0.22 µm nylon membranes (Millipore, USA). One µL of solution was then applied on a silica gel 60 TLC plate (Merck Darmstadt, Germany), with aluminum support. TLC plates were developed in a butanol/ propanol/ water (3:12:4) solvent system, and carbohydrates were revealed using aniline/ diphenylamine/ phosphoric acid in acetone.

Ion Chromatography

Analytical procedure was based on the method outlined in (Mellado-Mojica et al., 2017, 2022). The carbohydrate profiles of all samples were examined using HPAEC-PAD on a Dionex DIONEX ICS-3000 ion chromatograph (Thermoscientific, USA). The system was fitted with a Carbowax PA-200 (40 × 250 mm) column and a precolumn (40 × 25 mm).

Before injection, the samples were diluted with deionized water (resistivity of 17 MΩ) to a concentration of 10 mg mL⁻¹ and then filtered through 0.22 µm nylon membranes (Millipore, USA).

The separation gradient for the samples consisted of NaOH [0.23 M], CH₃COONa [0.5 M], and water, with a constant flow rate of 0.2 mL min⁻¹.

The sample separation gradient included NaOH [0.23 M], CH₃COONa [0.5 M], and water, with a constant flow rate of 0.2 mL min⁻¹. The separation began with a gradient of 70% water (A), 30% aqueous NaOH (B),

and 0% NaOH/CH₃COONa (C). The subsequent percentual ratios of A and B were as follows: 50:50 (5 min), 45:40 (8 min), 40:30 (27 min), 30:20 (15 min), 15:10 (10 min), and 50:50 (10 min). When the sum of A and B was less than 100%, the remainder of the mobile phase was composed of C. The compounds were detected using an amperometric detector, with the following potentials for amperometric detection: +0.1, -2.0, +0.6 y -0.1 V for E1 (400 ms), E2 (20 ms), E3 (20 ms), and E4 (60 ms), respectively.

Results and Discussion

Persimmon powder (labelled as 1) and syrup (labelled as 3) are depicted in Figure 1. It is worth mentioning that in the picture are also confectioneries (chocolate paste) prepared with both persimmon powder and persimmon syrup (2 and 4, respectively) but preparation of confectioneries from persimmon extracts is a work in progress, thus, it is beyond the scope of this work.

Figure 1

Persimmon extracts where 1: Persimmon powder. 2: Chocolate paste made from powder 3: Persimmon syrup. 4: Chocolate paste made from syrup

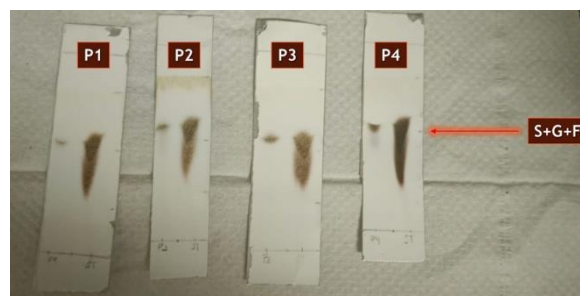


The results of SPME-GC-MS analyses are displayed in Table 1. We evaluated the volatile profile of persimmon extracts (both powder and syrup) to assess their potential as ingredients in confectionery products. Since the volatile profile is closely associated with aroma, we aimed to identify the aromatic compounds present. 19 compounds were identified in the powder whereas only 7 were identified in the syrup. This could be explained by the fact that in the syrup, part of the material (the bagasse) was discarded. Additionally, the powder was exposed to higher temperatures, which may have led to the formation of compounds resulting from thermal decomposition. The most abundant compound in both cases is furfural (32.12% in powder, and 67.56% in syrup). Furfural has been widely used as flavour enhancer for foods and drinks, since its smell is described as 'Almond, Caramel, Sweet, Woody' with a 'pleasant and aromatic odour', albeit its threshold is

relatively high thus it may not be recognized if used in a confectionery product (Eseyin & Steele, 2015; Rowe, 2004). Although it may be toxic for dogs, its toxicity for humans is low (Eseyin & Steele, 2015), since it is reported to have 'none' in all of the following categories: genotoxicity, repeated dose toxicity, reproductive toxicity, skin sensitisation, photoirritation/photoallergenicity, environmental toxicity, and read-across justification (Api et al., 2024). Furfural has also been used as fungicide and nematocide (Eseyin & Steele, 2015). 3-phenylindole is the next abundant compound in both persimmon powder and persimmon syrup. It has been regarded as an antimicrobial compound (Sinha & Smejtek, 1983). The sum of furfural and 3-phenyl indole account for little over half of the relative abundance of volatile compounds in the case of persimmon powder (51.3%) whereas they account for the vast majority of the relative abundance of volatile compounds in persimmon syrup (89.38%). In a study by Yang and colleagues in 2021, it was demonstrated that persimmon polysaccharide extracts had positive effects on the proliferation of lactobacilli (Yang et al., 2021). Although the authors did not report the presence of fructans, fructose polymers with well-documented prebiotic effects that favour lactobacilli growth (Mellado-Mojica et al., 2022; Mellado-Mojica & López, 2015), we aimed to measure the carbohydrate profile to evaluate the potential presence of fructans. For this purpose, we first employed TLC and then IC, using a standard composed of glucose, fructose, sucrose, and fructooligosaccharides (FOS). Results of TLC are displayed in Figure 2.

Figure 2

TLC plates of persimmon extracts where: P1: Persimmon powder, P2: Chocolate paste made from powder. P3: Persimmon syrup, P4: Chocolate paste made from syrup. S: Sucrose, G: Glucose, and F: Fructose



As mentioned in relation to Figure 1, there are also confectioneries (chocolate paste) prepared with both persimmon powder and persimmon syrup (P2 and P4, respectively) but preparation of confectioneries from persimmon extracts is a work in progress thus it is beyond the scope of this work. Although it can be seen in Figure 2 that no saccharide beyond S+G+F is present in either persimmon extract, we decided to use a more powerful technique to assess potential fructans.

Results of IC are displayed in Figure 3. Although IC analyses were not done quantitatively, it is still possible to confirm that carbohydrate profile of both persimmon powder and persimmon syrup is comprised of sucrose-fructose-glucose whereas little to no FOS are detected. Those results are in concordance with Yang et al. since they reported a carbohydrate composition of 98.2% glucose (Yang et al., 2021).

Figure 3

Ion chromatograms of persimmon extracts where: A: Standard, B: Powder, C: Syrup. Retention times for S+G+F and for FOS are indicated

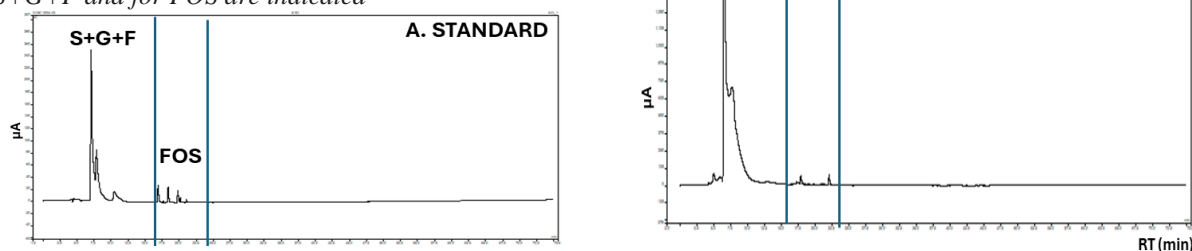


Table 1

Volatile compounds in persimmon extracts (powder and syrup) as analysed by SPME-GC-MS (values in % of total chromatogram area). C.V.: Coefficient of Variation (values in % of area percentage for each reported compound)

RT (min)	Compound	Persimmon powder (%)	C.V. (%)	Persimmon syrup (%)	C.V. (%)
17.192	Butanal	1.70	6.31		
17.489	Furfural	32.12	0.42	67.56	1.34
18.232	3-Phenylindole	19.19	0.90	21.82	1.65
18.576	N-(3-Methylphenyl)-4-(3-pyridinyl)-1,3-thiazol-2-amine	1.64	3.34		
18.745	1,5-Diphenyl-3-methylthio-1,2,4-triazole	5.73	0.82		
19.039	Benzaldehyde	0.15	6.17	0.92	5.71
20.124	2-Furancarboxaldehyde, 5-methyl-	0.27	4.89	1.84	2.21
20.728	Tetradecyl 2-methoxyacetate	1.95	0.42		
21.884	Benzeneacetaldehyde			0.22	4.21
22.231	1-Propanol, 3,3'-oxybis-	3.43	2.17		
22.243	Methane, diethoxy-	4.58	1.21		
22.28	2-Propanol, 1-[1-methyl-2-(2-propenyloxy)ethoxy]-	2.40	4.05		
22.387	3-Methyl-hexanoic acid			8.60	0.69
22.496	4,8,12,16-tetraoxaeicosan-1-ol	9.44	0.75		
23.919	Benzaldehyde, 3,4-dimethyl-	1.86	2.47		
23.928	Benzaldehyde, 4-ethyl-	1.62	3.31		
24.245	6-Tridecene, 7-methyl-	4.97	1.03		
26.224	Hexanoic acid	0.44	3.59		
26.243	Propyl octanoate	0.14	5.31		
28.319	3-Buten-2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-	6.25	1.18		

RT (min)	Compound	Persimmon powder (%)	C.V. (%)	Persimmon syrup (%)	C.V. (%)
30.364	2-Quinolinecarboxylic acid, methyl ester			0.61	4.12
31.254	Ethanol, 2-[2-(2-ethoxyethoxy)ethoxy]-	2.23	3.41		

Conclusions

1. The most abundant compound in the volatile profiles of both persimmon powder and persimmon syrup was furfural, a compound commonly used as a flavouring agent in the food industry. This highlights the potential of persimmon derivatives as natural sources of desirable flavour compounds.
2. The sugar profiles of persimmon powder and syrup revealed that most of their carbohydrates consist of simple sugars, including glucose, sucrose, and fructose, with little to no fructans detected.
3. The results demonstrate that persimmon powder and syrup could be used for the development of new, potentially more shelf-stable, confectionery products.

Incorporating these derivatives can simultaneously reduce persimmon waste and enhance the utilization of this highly perishable fruit. However, it is important to mention that shelf-life studies should be performed in confectionery products made from both persimmon extracts.

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