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ARAŞTIRMA MAKALESİ **RESEARCH ARTICLE**

A comprehensive study on determination of chemical composition of kumquat (*Fortunella margarita* L.) fruits

Kamkat (*Fortunella margarita* L.) meyvelerinin kimyasal bileşiminin belirlenmesi üzerine kapsamlı bir çalışma

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ABSTRACT

Kumquat (*Fortunella margarita* L.) is a group of small-sized fruits with a typical aroma. In this study, it was aimed to determine the chemical composition of kumquat fruit including β-carotene, sorbitol, sugars, organic acids and phenolics. According to High-Performance Liquid Chromatography (HPLC) analysis, β-carotene content was found to be 0.43 mg 100 g^{-1} in fresh fruit. Sucrose accounted for more than half of the sugar content with a ratio of 3.19 mg 100 g^{-1}. Citric acid was the most abundant organic acid in kumquat. A total of 14 phenolic compounds were investigated to reveal the phenolic composition of *F. margarita* and the results showed that it had a great diversity of phenolics. Gallic acid (277.3 mg 100 g^{-1}), procyanidin B1 (119.3 mg 100 g^{-1}) and epigallocatechin (89.3 mg 100 g^{-1}) were the major phenolic compounds identified in the whole fruit. As the fruit was eaten without peeling, the essential oil of the peel (PEO) was also analyzed by Gas Chromatography-Mass Spectrometry (GC-MS). It was determined that the PEO was a mixture of monoterpenes, sesquiterpenes, oxygenated terpenic compounds, esters and aldehydes. Limonene (83.8%), a monoterpenic substance, was the major aromatic component in the PEO, followed by β-myrcene (6.7%), germacrene D (3.3%) and α-pinene (1.8%). This work has shown that kumquat fruit is a natural source for bioactive compounds.

ÖZET

Kamkat (*Fortunella margarita* L.) tipik aromaya sahip küçük boyutlu bir meyve grubudur. Bu çalışmada, kamkat meyvesinin β-karoten, sorbitol, şekerler, organik asitler ve fenolikleri içeren kimyasal bileşiminin belirlenmesi amaçlanmıştır. Yüksek Performanslı Sıvı Kromatografisi (HPLC) analizine göre, taze meyvede β-karoten içeriği 0.43 mg 100 g^{-1} olarak bulunmuştur. Sakkaroz, 3,19 mg 100 g^{-1} oranı ile şeker içeriğinin yarısından fazlasını oluşturmuştur. Sitrik asit kamkatta en bol bulunan organik asit olmuştur. *F. margarita*'nın fenolik bileşimini ortaya çıkarmak için toplam 14 fenolik bileşik araştırılmış ve sonuçlar meyvenin büyük bir fenolik çeşitliliğine sahip olduğunu göstermiştir. Gallik asit (277.3 mg 100 g^{-1}), prosiyanidin B1 (119.3 mg 100 g^{-1}) ve epigallokateşin (89.3 mg 100 g^{-1}) bütün meyvede tespit edilen başlıca fenolik bileşiklerdir. Meyve soyulmadan yenildiği için, kabuk uçucu yağı (PEO) da Gaz Kromatografisi-Kütle Spektrometresi'nde (GC-MS) analize tabi tutulmuştur. PEO'nun monoterpenler, seskiterpenler, oksijenli terpenik bileşikler, esterler ve aldehitlerin bir karışımı olduğu belirlenmiştir. Monoterpenik bir madde olan limonen (%83.8) PEO'daki başlıca aromatik bileşen olup, bunu β-mirsen (%6.7), germakren D (%3.3) ve α-pinen (%1.8) takip etmektedir. Bu çalışma, kamkat meyvesinin biyoaktif bileşikler için doğal bir kaynak olduğunu göstermiştir.

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INTRODUCTION

Kumquat, *Fortunella* spp., is known as the smallest citrus fruit and differs from others in that the fruit is eaten with the peel (Schirra et al., 2008; Güney et al., 2015; Zeng et al., 2023). The *Fortunella* genus is closely related to *Citrus* and both belong to Rutaceae family (Çakmakçı et al., 2016; Tan et al., 2016; Sicari & Poiana, 2017). It mainly consists of six major cultivated species including *F. margarita, F. japonica, F. crassifolia, F. obovate, F. polyandra* and *F. hindsii*. Kumquat is native to China and its name means 'gold orange' in Chinese. The fruit can be varied round or oval-shaped depending on the species (Palma & D'Aquino, 2018). Kumquat is an excellent source of nutrients and phytochemicals (Zeng et al., 2015; Palma & D'Aquino, 2018). It has a wide variety of phenolic groups including flavonols such as rutin, quercetin (Chen et al., 2021) and kaempferol (Pawelczyk et al., 2023); flavanones such as narirutin and naringin (Chen et al., 2021; Pawelczyk et al., 2023); phenolic acids such as p-coumaric acid and ferulic acid (Lou & Ho, 2017). In previous studies, total phenolic content was reported to be between 98.6-3000 mg gallic acid eq. 100 g^{-1} depending on the fruit variety, the part of the fruit used and the extraction procedure (Lou et al., 2016; Perez, 2022; Li et al., 2023). Moreover, its total phenolic content in the peel was found to be higher than in lemons and in some cases even higher than in pummelos, oranges and mandarins (Chen et al., 2021). Studies have shown that kumquat extracts exhibit high antioxidant activity due to their radical scavenging capacity and ferric reducing power (Lou et al., 2016; Tan et al., 2016). The extracts have been determined to be effective in the prevention of various life-threatening diseases, especially cancer (Jayaprakasha et al., 2012), hyperglycemia, dyslipidemia (Tan et al., 2014), cirrhosis and aging-related diseases (Li et al., 2023). Kumquat consumption has also been noted to have positive effects in preventing or reducing of some hematal implications such as blood vessel rupture, arterial hardening and blood capillary fragility (Zeng et al., 2015). The fruit is commonly used in traditional medicine applications to treat respiratory tract inflammation, cough and cold (Tan et al., 2016; Lou & Ho, 2017). These health-promoting effects are mainly attributed to phenolic and flavonoid substances in the fruit (Li et al., 2023; Pawelczyk et al., 2023). However, the bioactive compounds are not only limited to phenolics, but essential oils, which are a natural part of the peel, also host a very large number of components. It was noted that monoterpenic compounds constitute the majority of the PEO composition (Lakache et al., 2022; Shalaby et al., 2023). As in other citrus species, limonene is the most abundant component in kumquat PEO (Küçükbay et al., 2025). β-myrcene and germacrene D were also found to be present in significant amounts in the PEO (Fitsiou et al., 2016; Shalaby et al., 2023). The other volatiles also play a crucial role in the flavor and biological activity of kumquat, even in minor concentrations (Ziogas et al., 2024). It has antibacterial activity on some pathogenic bacteria as well as being a remarkable antifungal agent (Wang et al., 2012). The PEO also contributes to fruit's anti-inflammatory and radical scavenging activities (Lakache et al., 2022). Thus, the medicinal and pharmaceutical industries are also interested in kumquat fruit because of its health-promoting effects (Zeng et al., 2015).

Despite its biological activity and health benefits, the number of comprehensive studies on fruit composition is limited. In particular, the diversity of phenolics in the fruit has not been fully elucidated, yet. Researchers working with kumquat are forced to refer to other *Citrus* fruits due to a lack of knowledge. Even though the genus *Fortunella* is closely related to the genus *Citrus*, they are quite different from each other in terms of chemical composition (Tan et al., 2016). In this study, it was aimed to determine β-carotene, organic acid, sugar, sorbitol and phenolic content of kumquat (*F. margarita*) fruits. As the fruits are eaten unpeeled, the peel essential oil (PEO) was also investigated for its volatile constituents.

MATERIALS and METHODS

Fruit material and sample preparation

Fresh kumquats (*F. margarita* Lour. Swingle cv. ovale) at commercial maturity were harvested from the gardens located in Mersin province (Southern Turkey; latitude 36° 37' 56.3'' N, longitude 34° 20' 37.6'' E, altitude 10 m) in December. Taxonomic identification of the fruits was carried out by Dr. Filiz Baysal (Alata Horticultural Crops Research Institute, Mersin) as Nagami kumquat SRA169. Fresh fruits were immediately transferred to department laboratories using cold-chain procedures. Fresh fruits were washed and halved to remove seeds. The pulp (including peel) following after the homogenization was subjected to HPLC analysis to determine the composition of phenolics, β-carotene, organic acid, sugar and sorbitol.

To identify the volatiles in PEO, the fruit was carefully peeled off and the peel material was shredded. 100 g of peel was weighed into a 2 L round flask containing 500 mL of distilled water. The flask was boiled with a mantle and distillation was performed using an all-glass Clevenger apparatus. Required time of hydrodistillation process was optimized as 3.5 hours considering the essential oil yield. The PEO obtained in each cycle was then combined, and subjected to anhydrous Na_2SO_4 in order to remove any residual water droplets in the oil phase (Abd-Elwahab et al., 2016). Samples were taken to amber coloured and sealed vials for determination of the volatiles. Specific gravity and refractive index were determined at 20°C (Shalaby et al., 2023). The yield of PEO was assessed according to the following formula (1) (Yu et al., 2021):

$$Y\,(\%) = (V/m) \times 100 \qquad (1)$$

where Y is the percentage of the essential oil yield; V is the obtained volume (mL) of essential oil; m (g) is the mass of kumquat peel subjected to the hydrodistillation process.

HPLC analysis

The phenolic composition, β-carotene, organic acid, sugar and sorbitol content of the fruit were determined by Shimadzu HPLC (Kyoto, Japan) coupled with an autosampler (SIL20A HT). The system was directly integrated with an oven (CTO-10AS VP), a degasser unit (DGU2A 5R), a gradient pump (LC-20AR) and detector systems (SPDM 20A, RID-10A). All chemicals and analytical standards were of chromatographic grade (>%99.9) and were purchased from Sigma-Aldrich (Germany).

β-carotene

Extraction, detection and quantification of β-carotene were conducted according to the procedures mentioned by Karabulut et al. (2018). 5 g of kumquat pulp was well mixed with 20 mL of 0.1% butylated hydroxytoluene (BHT in hexane:acetone:ethanol, 2:1:1, v:v:v). Obtained suspension was centrifugated at 6000×*g* for 5 min (Thermo Fisher Scientific, MA, USA). Three phases were distinctly separated from each other: pellet, polar and non-polar supernatants. Non-polar layer of the supernatant was transferred to a volumetric flask (25 mL). Remained mixture was re-extracted twice with 5 mL of hexane. Extracts were gathered together and the missing volume was made up to 25 mL with same solvent. 1 mL of extract was taken to an HPLC vial and the solvent was evaporated under nitrogen gas flow. Residual fragments were reconstituted in 1 mL tetrahydrofuran:methanol (1:9, v:v) containing 0.1% BHT and passed through a 0.45 µm PTFE filter.

Chromatographic separation of β-carotene was carried out on ODS-2 column (250×4.6 mm, 5 mm; GL Sciences Inc.; Japan) under isocratical conditions. Methanol:acetone (70:30, v:v) solution was used as mobile phase. Analysis conditions including injection volume, flow rate and column temperature were set as 20 µL, a flow rate of 1.0 mL min^{-1}, at 35 °C, respectively. β-carotene was monitored at 450 nm.

Sugars, sorbitol and organic acids

Sugar, sorbitol and organic acid analysis were carried out by the method stated by Ünal et al. (2023) with minor modifications. Briefly, 2 g of fresh fruit pulp was diluted up to 10 mL ultrapure water and the mixture was centrifugated at 6000×*g* for 5 min. The extract was filtered through a 0.45 µm nylon filter and 20 µL of the sample was injected into HPLC column. Chromatographic separation was achieved isocratically on Rezex column (300×7.8 mm, Phenomenex; CA, USA). For sugar and sorbitol analysis, the flow rate of mobile phase (ultrapure water) was set as 0.6 mL min^{-1}, at 80 °C. Sugars and sorbitol were monitored by using a refractive index detector. For the analysis of organic acids, a mobile phase consisting of 2.5 mM H_2SO_4 was used. Elution was programmed at a flow rate of 0.5 mL min^{-1}, at 55 °C. In PDA detector system, ascorbic acid was monitored at 245 nm while malic and citric acids were at 210 nm.

Phenolic compounds

Approximately 20 g of fruit pulp was vigorously mixed with 50 mL of methanol:water (40:10, v:v) supplemented with 0.4% ascorbic acid. The headspace of the tubes was then filled with nitrogen gas and placed in an ultrasonic bath for 20 min. Samples were clarified by centrifugation at 6000×*g*, for 5 min and the organic phase was carefully collected. The process was repeated twice using 15 mL of extraction solution. The clarified liquid phases were combined in a flask and the deficient volume was made up to 100 mL with the extraction solution (Karabulut et al., 2018).

Analysis of phenolic compounds was performed on HPLC system using an ODS-3 column (250×4.6 mm, 5 µm; GL Sciences Inc., Japan) according to the procedures described by Campbell et al. (2013). 20 µL of extract was injected into the column and oven temperature was maintained at 25 °C throughout the analysis. Ultrapure water (A) and acetonitrile (B), both containing 0.1% phosphoric acid, were run at a total flow rate of 1 mL min^{-1} for elution. The gradient programme based on phase B was set as follows: ramp from 8% to 11% for 4 min, reach to 35% at 25 min, 60% at 30 min, hold at 60% for 10 min; and then decrease to 35% at 45 min, 8% at 50 min and hold at 8% for 5 min. PDA detector system was used for monitoring the phenolic compounds at given wavelengths: 280 nm for gallic acid, catechin, epicatechin, epigallocatechin, procyanidin B1 and B2; 320 nm for caffeic acid, ferulic acid, p-coumaric acid, chlorogenic acid and neo-chlorogenic acid; 360 nm for quercetin 3-β-D-glucoside, kaempferol-3-glucoside and rutin hydrate.

Method validation

The proficiency of the HPLC methods was characterized by in-house analysis of standard solutions, spiked and unspiked samples. Quantification was done using a six-point calibration curve according to the external standard method. The working range of the standard solutions is given in Table 1. Linearity was determined by the calibration curve generated after three injections of each level of standard solutions. Accuracy was assessed by calculating the average recovery rates from spiked samples according to the standard addition method. For the purpose, samples with known analyte concentrations were spiked with the corresponding analytical standard at three levels: the lowest, intermediate and the highest concentrations in the calibration series. The recovery rate was determined by calculating the ratio of the amount of standard detected (after subtracting initial analyte amount) to the amount of standard spiked (ICH, 2005; da Silva Padilha et al., 2017; Chen et al., 2021).

Repeatability (RSD_r%) and precision (RSD_R%) were assessed by referring the relative standard deviation. The samples spiked with analyte at intermediate concentration were analyzed by same operator for three consecutive days (intermediate precision, n=18) with six repetitions per day (repeatability, n=6) (ICH, 2005; Chen et al., 2021). The relative standard deviation (RSD) was attained by calculating the ratio of the standard deviation to the mean. Since blank sample matrices were not available for the compounds studied, mix standard solutions were used to determine theoretical limit of detection (LOD) and limit of quantification (LOQ). The analytical standards were prepared at concentrations close to the estimated LOD. After ten replicate analyses, LOD and LOQ were calculated by 3 and 10 times the standard deviation, respectively (Natividade et al., 2013; Chen et al., 2021).

GC analysis of volatiles

Shimadzu QP-2010 Plus GC (Japan) integrated with MS/QP 2010 was used for identification of volatile constituents in PEO. Samples were injected into a DB-WAX (J&W Scientific, Folsom, CA) capillary column with a length of 60 m, an inner diameter of 0.25 mm and a film thickness of 0.25 µm. 1 µL of sample in hexane (1:10, v:v) was injected into the port by an AOC-20i/20s autosampler with a split ratio of 1:100. High purity helium (>99.99%), the carrier gas, was flowing constantly at 1 mL min^{-1}. The column oven was conditioned by applying a temperature gradient. The initial temperature was maintained at 40 °C for 5 min, followed by a ramp of 3 °C per minute to 240 °C and kept at that temperature for 15 min. The injection port and line temperatures were set at 250 °C. The MS detector was run with a 70 eV ionization source in EI mode. Ions were scanned at a rate of 1 scan s^{-1} and the mass spectra were obtained for the ions with mass-to-charge ratios ranging between 45 and 450 (Karakaya et al., 2022).

Identification of the substances was achieved by matching the mass fragmentation profiles with those of commercial libraries installed on the instrument. In addition, they were confirmed by comparison of RI values in literature records compiled from analysis performed under identical conditions (Wallace, 2021). A homologous series of *n*-alkanes (C8–C20 and C21–C40) was used as benchmarks for determining retention indices (RI) (Adams, 2007). D-limonene (>97%; Sigma-Aldrich, Germany), which has been reported as the predominant essential oil component in citrus fruits, was used as a reference material to verify the retention time, retention indice and mass fragmentation of the related peak. Semi-quantification was done according to area normalization. GC-MS analyses were carried out in duplicate.

Statistical analyses

All HPLC analyses were conducted in triplicate. The results are presented as mean ± standard deviation. One-way analysis of variance (ANOVA) and Tukey's HSD tests were performed using IBM SPSS Statistics ver. 25 (SPSS Inc., IL, USA) software. Differences between the means were considered significant at 5% (p <0.05) level.

RESULTS and DISCUSSIONS

Proficiency of HPLC methods

The results related with performance analyses of HPLC methods are summarized in Table 1. An R value greater than 0.99 indicates that calibration curve is linear over the operating range (da Silva Padilha et al., 2017; Coelho et al., 2018). The correlation coefficients (r^2) were found to range from 0.9911 to 0.9999 for all analytes tested, demonstrating that these calibration plots could be used in quantification. The LOD and LOQ values were quite low in the study. It implied that the analytical methods used were enough sensitive to reveal the chemical composition of kumquat in terms of the compounds examined. Repeatability and precision were evaluated by replicated analyses of spiked samples within a day and between days, respectively. The overall RSD% for both intra- and inter-days were less than 5% and it concluded that the proposed methods exhibit acceptable repeatability and adequate precision.

Table 1. Retention times, linearity ranges, equations and R^2 values of the components examined
Çizelge 1. İncelenen bileşenlerin alıkonma süreleri, doğrusallık aralıkları, denklemleri ve R^2 değerleri

Component	RT* (min)	Range**	r^2	LOD	LOQ	Repeatability, RSD_r (%)	Precision, RSD_R (%)
β-carotene	18.66	0.625 – 20 Y = 8.5909 X + 0.1483	0.9998	0.674	2.247	0.25	0.41
Glucose	10.96	0.625 – 20 Y = 3.7906 X - 0.0374	0.9995	0.055	0.183	2.80	3.46
Fructose	15.23	0.625 – 20 Y = 4.1176 X + 0.0465	0.9998	0.046	0.153	2.24	1.89
Sucrose	9.50	0.625 – 20 Y = 3.8117 X - 0.0304	0.9998	0.011	0.037	1.31	1.65
Sorbitol	26.13	0.625 – 20 Y = 3.9966 X + 0.0585	0.9997	0.042	0.140	2.28	2.62
Malic acid	13.17	0.25 – 8 Y = 5.3019 X - 0.0019	0.9999	0.008	0.027	3.88	4.36
Citric acid	11.08	0.25 – 8 Y = 3.9512 X + 0.0183	0.9998	0.001	0.002	1.94	2.52
Ascorbic acid	13.84	0.25 – 8 Y = 7.9861 X + 0.0149	0.9997	0.007	0.022	2.46	2.88
Gallic acid	6.77	0.5 – 20 Y = 2.0579 X +0.2470	0.9933	0.101	0.337	2.70	2.48
Procyanidin B1	12.02	0.5 – 20 Y = 9.4192 X + 0.2825	0.9966	0.084	0.280	2.15	2.76
Epigallocatechin	12.35	1 – 40 Y = 0.0004 X + 2.3611	0.9911	0.076	0.253	2.54	3.02
Catechin	14.10	1 – 40 Y = 7.0454 X + 0.5504	0.9978	0.107	0.357	2.88	3.46
Procyanidin B2	15.04	0.5 – 20 Y = 0.0001 X - 0.3029	0.9963	0.057	0.190	2.26	2.59
Epicatechin	16.19	1 – 40 Y = 4.8955 X + 0.5060	0.9976	0.063	0.210	2.53	3.18
Neo-chlorogenic acid	10.83	1 – 40 Y = 1.6916 X - 0.0980	0.9981	0.044	0.147	2.43	2.20
Chlorogenic acid	13.82	1 – 40 Y = 1.1545 X - 0.0272	0.9978	0.037	0.123	3.15	3.76
Caffeic acid	16.38	0.5 – 20 Y = 5.5661 X - 0.0975	0.9992	0.096	0.320	4.03	4.68
p-coumaric acid	20.60	1 – 40 Y = 7.9960 X - 0.1012	0.9973	0.086	0.287	1.48	1.88
Ferulic acid	21.78	0.5 – 20 Y = 7.5258 X - 0.1172	0.9989	0.062	0.207	3.67	3.24
Rutin hydrate	19.47	1 – 40 Y = 5.4039 X + 0.5343	0.9977	0.035	0.117	2.29	2.74
Quercetin 3-*β*-D-glucoside	20.73	0.5 – 20 Y = 2.9906 X + 0.2382	0.9977	0.046	0.153	2.39	2.65
Kaempferol-3-glucoside	22.66	0.5 – 20 Y = 3.8905 X + 0.2927	0.9961	0.054	0.180	2.34	2.86

* RT, retentin time; LOD, limit of detection; LOQ, limit of quantification.
** The concentrations are mg kg^{-1} for β-carotene and phenolics; mg g^{-1} for sugars, sorbitol and organic acids.

Recovery rates of β-carotene, sugars, sorbitol, organic acids and phenolics ranged between 94.4-98.7%, 91.7-105.6%, 87.5-94.8%, 90.7-108.7% and 81.3-104.0%, respectively (Fig. 1). In general, the rates between 80 and 110% are considered satisfactory for validation studies (Natividade et al., 2013). Some past studies on fruits and juices have reported recovery rates between 70 and 110% for these compound groups (Scherer et al., 2012; Natividade et al., 2013; da Silva Padilha et al., 2017; Coelho et al., 2018). The present results were in line with previous reports and the recovery rates were considerably high, indicating the accuracy of the method.

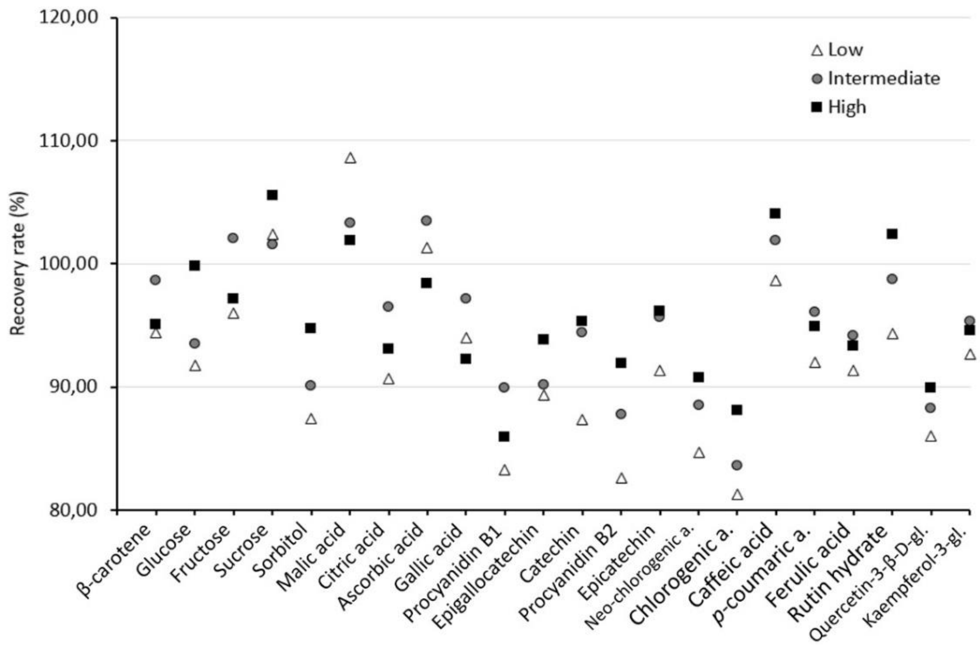

Figure 1. Recovery rates obtained from samples spiked with three different concentrations of analytes

Şekil 1. Üç farklı konsantrasyonda analit eklenmiş numunelerden elde edilen geri kazanım oranları

Chemical composition of kumquat fruits

Carotenoids are mainly located in the kumquat peel while the juice contains small amounts (Agócs et al., 2007). Many citrus fruits contain less than 0.05 mg 100 g-1 of β-carotene in the pulp (Agócs et al., 2007; Wang et al., 2007; Sun et al., 2018). In this work, it was determined to be 0.43±0.02 mg 100 g-1 fw in the peel and pulp of kumquat fruits. Kumquat was found to have a higher β-carotene content than the content reported in its relatives in the Citrus genus. In previous researches, it was found to vary between 0.33-0.45 mg 100 g-1 for different kumquat cultivations, and the present result has supported these findings (Schirra et al., 2008; Souza et al., 2021). It is thought that the peel material may have contributed significantly to the high β-carotene content in the pulp.

The sugar, sorbitol and organic acid contents of the fruits are shown in Table 2. The amount of sucrose was slightly higher than the sum of glucose and fructose, in agreement with previous studies (Schirra et al., 2008; Wei et al., 2021). Similar results were also obtained in kumquats grafted onto different Citrus rootstocks (Babazadeh & Jaimand, 2019). Kumquat is evaluated as a fruit with a low glycemic index due to its low sugar content and the inclusion of fiber in its peel in the diet. As a result of these properties, it has been reported to aid weight loss (Karahuseyin & Nenni, 2023; Pawelczyk et al., 2023) and reduce fasting plasma glucose levels to control values (Massaro et al., 2025). On the other hand, the present results were found to be relatively low in terms of total sugar content compared to previous findings (Shanmugavelan et al., 2013; Wei et al., 2021). Due to the global distribution of cultivars, compositional diversity is inevitable in kumquats grown in different regions of the world. Even within the same species, compositional differences can be observed, both in terms of nutritional and bioactive components. This is due to several factors such as genetic structure, fruit maturity, geographical and climatic conditions, post-harvest handling processes, cultural management and the use of non-standardized extraction and analytical methods (Schirra et al., 2008; Palma & D'Aquino, 2018). However, it has also been stated that temperature conditions are quite effective on sugar accumulation. Warmer seasons cause a decrease in the soluble sugar content of the fruit (Chang & Lin, 2020). One or more of above factors may have affected the sugar composition of the kumquats. Sorbitol was not detected in the samples, indicating that it was not present in either the peel or the juice.

Table 2. Sugar, sorbitol and organic acid contents of the kumquat fruits
Çizelge 2. Kamkat meyvelerinin şeker, sorbitol ve organik asit içerikleri

Group	Compound	Amount (mg 100 g^{-1} fw)
Sugars	Glucose	829.89 ± 50.24^{a}
	Fructose	2136.65 ± 111.20^{b}
	Sucrose	3184.98 ± 183.27^{c}
Sugar Alcohol	Sorbitol	ND*
Organic Acids	Malic acid	187.16 ± 6.70^{a}
	Citric acid	2402.62 ± 57.45^{b}
	Ascorbic acid	14.35 ± 0.07^{c}

* ND, not detected.

Citric acid is referred to as the major contributor of organic acids in various *Citrus* fruits (Asencio et al., 2018; Cano-Lamadrid et al., 2018; Zhou et al; 2018; Liao et al., 2019). Its content was reported between 5.4-6.8 g 100 g^{-1} in sour lemon and lime cultivars, while it was about 0.11-1.35 g 100 g^{-1} in sweet lemon and lime cultivars (Asencio et al., 2018; Cano-Lamadrid et al., 2018). In other citrus fruits such as mandarin, sweet orange, pummelo and grapefruit, it was documented that citric acid content could exceed 1.5 g 100 g^{-1}, but dropped below 1 g 100 g^{-1} with ripening (Zhou et al; 2018). Even though kumquat contained moderate levels of citric acid (Table 2), it dominated the organic acid composition of the fruit, just as in its relatives in the genus *Citrus*. The presence of ascorbic acid is also very important due to its invaluable role for human body. Acting as an antioxidant, it prevents the occurrence of numerous diseases, including those caused by free radicals (Karahuseyin & Nenni, 2023). However, citrus varieties are not actually the richest fruits in terms of vitamin C. Orange, lemon, tangerine, grapefruit and kumquat varieties generally contain less than 60 mg 100 g^{-1} fw ascorbic acid. (Berk, 2016). In past reports, the total amount of ascorbic acid in kumquat peel and pulp ranged from 2.3-18.2 mg 100 g^{-1} fw (Schirra et al., 2008; Souza et al., 2021). The result obtained (14.35±0.07 mg 100 g^{-1} fw) was in agreement with those given in the literature. It has been seen that the vitamin C content in a 100 g portion of kumquat fruit can meet 36% of the recommended dietary allowance (40 mg day^{-1}) (Carr & Lykkesfeldt, 2021).

Organic acids and sugars are of interest because of their significant impact on the sensory characteristics of fruit juices (Asencio et al., 2018). Organic acids, especially citric acid, are responsible for the sour taste in fruits (Cano-Lamadrid et al., 2018). High acid levels often adversely affect fruit quality, but a moderate acid concentration can enhance the sensory acceptability of fruit (Liao et al., 2019). Because, citric acid levels balanced with sugars give the fruit a perception of sweetness (Cano-Lamadrid et al., 2018). Although it can be eaten along with the peel, kumquat is considered to have a slight sourness that is not dominant. During fruit ripening, organic acid degradation and sugar accumulation occur simultaneously (Zhou et al., 2018; Liao et al., 2019). Glucose, fructose and sucrose are present in all type of citrus fruits but the principal one can vary depending on fruit variety (Asencio et al., 2018), environmental factors and enzymatic pathways during fruit ripening and storage (Liao et al., 2019). Fructose is of great nutritional importance as being the natural sugar with the highest degree of sweetness among mono- and disaccharides. In particular, balancing the acidity with sugars creates an appealing sweetness and flavor that facilitates the processing of fruit into various edible products such as marmalades (Asencio et al., 2018). The present findings have shown that kumquat has a low sugar and mild organic acid content. Thus, it has the potential to be used in a wide variety of food formulations without compromising the taste of the food.

Previous reports have only addressed the total phenolic content of the fruit and have been deficient in expressing phenolic diversity. This may be due to the challenge of determining the phenolics that are covalently bound to insoluble plant material (Lou & Ho, 2017; Al-Saman et al., 2019; Zhang et al., 2020). Free phenolic acids have not yet been reported, except p-coumaric and ferulic acid which are found in kumquat via ester bonds (Lou & Ho, 2017). In a past report, the total phenolic content was determined to be 290.6 mg gallic acid eq. 100 g^{-1} fw (Schirra et al., 2008). In another study, p-coumaric acid was reported as the most abundant phenolic acid in the fruit (Lou & Ho, 2017). Chen et al. (2021) found no ferulic acid in the fruit peel, while p-coumaric acid was at trace levels. The phenolic compounds found in the kumquats are listed in Table 3. Contrary to the literature, kumquat provided a rich source of phenolics in terms of diversity and quantity. Gallic acid was the most abundant phenolic in the whole fruit (peel and pulp) followed by procyanidin B1 and epigallocatechin. Ferulic acid contributed significantly to the phenolic composition, as opposed to previous findings. Since the peel plays a primary role in protecting the fruit against physical and chemical deterioration; it tends to contain more bioactive compounds than the pulp (Lou et al., 2016; Souza et al., 2021). Most of the phenolics are also located in the peel rather than in the juice (Lou et al., 2016; Pawelczyk et al., 2023) and releasing them into extract is highly dependent on extraction method (Al-Saman et al., 2019). In addition, phenolic content varies with the maturity stage of the fruit and the part subjected to extraction. It was stated that the total phenolic content of immature kumquat was 1.5-2 fold higher than mature peel and pulp extracts (Lou et al., 2016). All these factors may be the reason for controversial reports regarding the phenolic composition of the fruit.

Table 3. Phenolic composition of kumquat fruits grown in Turkiye
Çizelge 3. Türkiye'de yetişen kamkat meyvelerinin fenolik bileşimi

Wavelength	**Compound**	**Amount (mg 100 g^{-1} fw)**
280 nm	Gallic acid	277.31 ± 2.06[a]
	Procyanidin B1	119.25 ± 16.49[b]
	Epigallocatechin	89.29 ± 0.42[c]
	Catechin	18.80 ± 4.00[d,e,f]
	Procyanidin B2	0.13 ± 0.02[g]
	Epicatechin	30.12 ± 6.12[d]
320 nm	Neo-chlorogenic acid	ND*
	Chlorogenic acid	3.27 ± 0.00[f,g]
	Caffeic acid	ND
	p-coumaric acid	5.78 ± 0.50[e,f,g]
	Ferulic acid	19.71 ± 1.04[d,e]
360 nm	Rutin hydrate	ND
	Quercetin 3-*β*-D-glucoside	10.38 ± 0.04[e,f,g]
	Kaempferol-3-glucoside	14.12 ± 0.32[d,e,f,g]

* ND, not detected.

Citrus peels have been used for many years for their pharmaceutical effects. However, they have not become a part of the daily diet because of their undesirable bitter taste (Tan et al., 2014). Kumquat is interestingly differed from other citrus fruits via consuming unpeel. Therefore, the phenolics found in the peel can be taken with the daily diet. For instance, p-coumaric acid and chlorogenic acid were found at levels of 0.26 and 0.18 mg g^{-1} in lemon (*C. limon* L. Bur.) peel, respectively (Lou et al., 2016). Discarding citrus peels causes loss in phenolics while they can be utilized in kumquat by eaten whole fruit. Polyphenolic compounds have a noteworthy impact on the nutritional value and health-promoting effects of fruits (Karahuseyin & Nenni, 2023; Pawelczyk et al., 2023). These substances are reported to prevent the formation of various diseases, especially cancer and aging-related disorders, by scavenging free radicals in the body (Pawelczyk et al., 2023). Lou et al. (2016) revealed a significant correlation between the total phenolic content of kumquat extracts and DPPH scavenging activity, which can be considered as an evidence for the antioxidant activity of phenolic compounds. In another study, kumquat extracts (100 µg mL^{-1}) obtained using different solvents showed significant inhibition on human prostate cancer. The anticancer properties of the ethanol-acetone extracts were attributed to the phenolic diversity of the fruit, while those of n-hexane extracts were associated with β-carotene and some volatile components in PEO (Jayaprakasha et al., 2012). Tan et al. (2014) determined that ethanolic extracts of kumquat fruits suppressed body weight gain and kept fasting blood glucose and serum lipid levels under control in mice fed a high-fat diet. Moreover, kumquat extracts improved glucose and insulin tolerance in obese mice, along with a decrease in serum lipid levels. The rich polyphenolic composition of kumquat, especially in the peel structure, has been evaluated to be effective in all these improvements seen in metabolic disorders. In order to exhibit biological activity, these substances must be accessible in the digestive tract and must be absorbed and transported to target tissues and organs. Unfortunately, there is no data on bioaccesibility and bioavailability of phenolics located in kumquat fruit matrix. According to a previous report using chemical approach, 62.4% of total polyphenolics of grape were found to be bioaccessible at the end of digestion (Tagliazucchi et al., 2010). In another *in vitro* digestion study, 75% of the total phenolic content of lyophilized sweet orange pulp was still found bioaccessible in the intestinal tract (Pena-Vazquez et al., 2022). In a contrast to high bioaccessibility rates of phenolics, their bioavailability remains very low. However, food polyphenols, regardless of their low bioavailability, have played an important role in protection against oxidative damage and cancer (Tagliazucchi et al., 2010).

Volatile substances in the peel essential oil

The PEO was clear liquid with a golden yellow colour and intense citrus fruity odour. The average essential oil yield of kumquat peel was determined as 1.38±0.16 % (v:w) on fresh weight. It has been stated that ripened kumquat fruits contain many oil glands in outer pericarp and PEO yield of the fruits varies between 1-2% (Zeng et al., 2023). The specific gravity and refractive index at 20°C were 0.843±0.01 g.cm^{-3} and 1.476, respectively.

A total of 35 aromatic compounds were identified in kumquat PEO by GC-MS analysis (Table 4). They were classified as monoterpenes (9), oxygenated monoterpenes (4), sesquiterpenes (12), oxygenated sesquiterpenes (2), esters (6), and aldehydes (2). Citrus essential oils are noted with terpenoid compounds and their oxygenated derivates (Shalaby et al., 2023). According to the results, monoterpenes represented 93.8% of total volatiles in kumquat PEO. D-limonene (83.84±1.43 %) was the major component, as in other *Citrus* essential oils (Sawamura, 2010). It is responsible for the fruit's characteristic citrus scent (Küçükbay et al., 2025). Its content was comparatively higher than that previously reported PEO for *F. crassifolia* (Wang et al., 2012). Furthermore, it has been stated that limonene content can reach over 92% in some *F. margarita* cultivars (Fitsiou et al., 2016; Yu et al., 2021; Shalaby et al., 2023). β-myrcene, germacrene D and α-pinene were also abundant in kumquat PEO, which was consistent with previous studies (Fitsiou et al., 2016; Lakache et al., 2022; Shalaby et al., 2023). Other volatile constituents may have also contributed to the flavour even if they were present in trace amounts.

Table 4. Volatile constituents of kumquat peel essential oil

Çizelge 4. Kamkat kabuk uçucu yağının uçucu bileşenleri

No	**Compound**	**RI_E***	**RI_L**	**%**
1	*α*-Pinene	1008	1013	1.80 ± 0.02
2	*β*-Pinene	1093	1094	0.05 ± 0.00
3	Sabinene	1107	1113	0.45 ± 0.00
4	*β*-Myrcene	1151	1160	6.72 ± 0.07
5	*D*-Limonene	1202	1200	83.84 ± 1.43
6	*β*-Phellandrene	1205	1201	0.88 ± 0.02
7	γ-Terpinene	1234	1240	0.04 ± 0.00
8	*β* -Ocimene	1238	1239	0.03 ± 0.00
9	*α*-Terpinolene	1270	1278	0.03 ± 0.00
10	Octyl acetate	1460	1461	0.21 ± 0.00
11	*α*-Copaene	1481	1488	0.04 ± 0.00
12	Decanal	1483	1481	0.08 ± 0.00
13	*β* -Cubebene	1526	1524	0.02 ± 0.00
14	Linalool	1530	1529	0.15 ± 0.00
15	*β* -Elemene	1576	1576	0.10 ± 0.00
16	*β* -Caryophyllene	1587	1585	0.06 ± 0.00
17	4-Terpineol	1588	1587	0.05 ± 0.00
18	*p*-Mentha-2,8-dien-1-ol	1610	1620	0.05 ± 0.00
19	Citronellyl acetate	1637	1644	0.02 ± 0.00
20	Humulene	1660	1663	0.04 ± 0.00
21	Decyl acetate	1666	1676	tr**
22	*α*-Amorphene	1677	1676	tr
23	*α*-Terpineol	1684	1683	0.11 ± 0.00
24	Germacrene D	1701	1702	3.31 ± 0.04
25	Neryl acetate	1707	1708	0.06 ± 0.00
26	α-Muurolene	1711	1716	tr
27	Bicyclogermacrene	1716	1706	0.62 ± 0.00
28	*α*-Farnesene	1729	1734	0.10 ± 0.00
29	Geranyl acetate	1740	1739	0.55 ± 0.03
30	*δ*-Cadinene	1744	1742	0.13 ± 0.00
31	Perilla aldehyde	1774	1776	0.10 ± 0.00
32	Germacrene B	1814	1816	0.16 ± 0.00
33	Limonen-10-yl acetate	1842	1846	0.05 ± 0.00
34	Nerolidol	2019	2023	0.06 ± 0.00
35	*α*-Cadinol	2220	2218	0.07 ± 0.00
	Aldehydes (No: 12, 31)			0.18
	Esters (No: 10, 19, 21, 25, 29, 33)			0.89
	Monoterpenes (No: 1-9)			93.84
	Oxygenated monoterpenes (No: 14, 17, 18, 23)			0.36
	Sesquiterpenes (No: 11, 13, 15, 16, 20, 22, 24, 26-28, 30, 32)			4.58
	Oxygenated sesquiterpenes (No: 34, 35)			0.13
	Total identified			**99.98**

* RI_E is experimentally calculated retention indice in the present study while RI_L refers to RI value which summarized from the literature and available in the NIST Chemistry Webbook (Wallace, 2021).

** Constituents measured below 0.01% were considered as trace (tr).

The composition of PEO can vary even between different varieties of the same species (Sawamura, 2010). Fruit maturity, cultivation and storage conditions, extraction and analysis methods used are other important factors affecting the essential oil composition (Zeng et al., 2023). As essential oils are complex mixtures, each compound contributes to the overall character of the essential oil (Tekeli et al., 2007). One of the most important features that distinguishes kumquat from other *Citrus* species is that the peel is also edible along with the flesh. This allows the compounds in PEO to directly influence the sensory (taste and flavour) and functional (health-promoting effects) properties of the fruit (Pawelczyk et al., 2023; Zeng et al., 2023).

In conclusion, kumquat is an important citrus fruit that has attracted the attention of consumers in recent years with its taste and appearance. Consuming the fruit as a whole increases the contribution and importance of the peel to the nutritional value. The present findings indicate that kumquat fruit serves as a significant source of bioactive compounds. β-carotene content of the fruit was relatively higher than the amount reported in many other citrus fruits before. Citric acid was determined as the dominant organic acid, as expected from *Citrus* and related fruit species. To our knowledge, this was the first comprehensive study to reveal the phenolic diversity of the whole fruit. Gallic acid was found to be the most abundant phenolic in the fruit, in contrast to some previous studies. The essential oil composition was also examined for its volatiles and it was determined that the majority of the oil composition consisted of terpenic compounds. Of these, D-limonene was found to be the main aromatic substance of the essential oil. In sum, the results of the study show that kumquat is a fruit with a relatively low sugar content, while being rich in phenolic and aromatic substances. It is currently consumed only in natural form or as marmalade but can be an alternative food to diversify to daily diet. Kumquat is considered to have potential for use in the development of health-promoting foods such as fortified beverages, snack bars, confectionery products, fruity yoghurts, ice-creams and dietary supplements. As consumer demand for natural and health-promoting products increases, kumquat may constitute a valuable source for innovative food formulations and it is recommended that further studies should focus on this issue.

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STATEMENT OF CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR'S CONTRIBUTIONS

HK: Investigation, Formal analysis, Supervision, Writing – Original draft, Writing – review & editing. TBK: Resources, Conceptualization, Methodology, Investigation, Formal analysis. FS: Investigation, Formal analysis, Writing – review & editing. KY: Investigation, Formal analysis.

STATEMENT OF ETHICS CONSENT

This article does not require ethical approval as there are no experiments with human or animal subjects.

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