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Essential Oil and Mineral Analysis of Citrus Peels

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Abstract

In this study, 5 different citrus fruits (mandarin, grapefruit, orange, kumquat and lemon) harvested in Turkey were analyzed qualitatively and quantitatively. For this purpose, essential oils obtained with the Clevenger apparatus. Essential oils components were determined with the help of GC-MS. Limonene was determined as a common component in all fruit peels, from the lowest to the highest in order, approximately 24% in clementine mandarin, 28% in orange and 76% in lemon, 98% in grapefruit, %100 kumquat. Linalool was determined main component of orange and clementine mandarin. Dry matter and ash of peels was determined. In addition, the elements found in the peels were determined with the help of semedx. According to the results of the sem-edx the peels are quite rich in terms of calcium and potassium.

Keywords: Mandarin, lemon, grapefruit, orange, kumquat

Narenciye Kabuklarinin Esansiyel Yağ ve Mineral Analizi

Özet

Bu çalışmada Türkiye'de hasat edilen 5 farklı narenciye (mandalina, greyfurt, portakal, kumkuat ve limon) kalitatif ve kantitatif olarak incelenmiştir. Bu amaçla Clevenger aparatı ile uçucu yağlar elde edilmiştir. Uçucu yağ bileşenleri GC-MS yardımıyla belirlendi. Limonen en düşükten en yükseğe doğru, clementine mandalinada yaklaşık %24, portakalda %28, limonda %76, greyfurtta %98 ve kumkuatta %100 oranda ortak bileşen olarak belirlenmiştir. Linalool, portakal ve mandalinanın ana bileşeni olarak belirlendi. Linalool, portakal ve mandalinanın ana bileşeni olarak belirlendi. Linalool, portakal ve mandalinanın ana bileşeni olarak belirlendi. Linalool, portakal ve mandalinanın ana bileşeni olarak belirlendi. Kabukların kuru maddesi ve külü belirlendi. Ayrıca kabuklarda bulunan elementler sem-edx yardımıyla belirlenmiştir. Sem-edx sonuçlarına göre kabuklar kalsiyum ve potasyum açısından oldukça zengindir. **Keywords:** Mandalina, limon, greyfurt, portakal, kumkuat

Introduction

Citrus peels are used in different industries such as food, cosmetics, pharmacy, perfumery and chemistry. Citrus peels, which are rich in phenolic acids and flavonoid glycosides, are also used in making pectin (Lee et al., 2022; Liu et al., 2022). When citrus peels are used in the right amounts, they improve the quality of the food by preserving its sensory properties. It is also stated that the peels have antioxidant, antimicrobial, antidiabetic, antihypertensive, anti-inflammatory and anticancer properties. Various research studies show that citrus peels are rich in vitamins, fiber and bioactive compounds. While essential oils or powder forms are used to benefit from the rich content of the peel in food products, it is still considered as waste in some parts of the (Ademosun, 2022; Liu et al., 2022). In a study, the antimicrobial effect of mandarin essential oil was tested and it was stated that it could be used as a natural antimicrobial agent in the food industry (Song et al., 2021). The antimicrobial effect of citrus peel extracts was tested against gram-positive and gram-negative bacteria, and the protective effect of sweet orange peel extract against oxidation of soybean oil and sunflower oil was determined (Shehata et al., 2021). It is stated that citrus peels show anti-inflammatory activity. It has been noted that citrus peels contain more bioactive components such as phenolic acids, flavonoids and limonoids and fiber than the juice of the fruit (Huang & Ho, 2010).

Dry matter (DM) is everything that remains in the fruit when all the water has been removed; this includes sugars, starch, cell walls, organic acids, fibers and minerals. It also provides information about fruit development maturity. Important for the quality of the fruit (Musacchi & Serra, 2018). Moisture content is a measure of the fruit's perishability (Li et al., 2019). Ash content in fruits constitutes the inorganic parts and these can be important elements in human nutrition such as Ca, P, K, Fe, Zn, Mg, S. Ash content is therefore a measure of the quality of food (Akinyele & Shokunbi, 2015; Chatfield & Adams, 1939). The peels dry matter and ash content analyses were done. Studies have shown that mandarin orange and lime peels are rich in K, Ca, Mg and Na (De Moraes Barros et al., 2012; Matsuo et al., 2019). 45% of the world's citrus production area is orange, 35% is mandarin, 15% is lemon and 4% is grapefruit. Turkey ranks seventh in world orange production with a production amount of 1.8 million tons, third in world mandarin production with 1.8 million tons, fourth in lemon production with 1.4 million tons, and fifth in world grapefruit production with 250 thousand tons of grapefruit production. Production is mostly done in the Aegean region after the Mediterranean region (Aygören, 2022). In addition to the abundance of these fruits being consumed, the fact that they grow in abundance in our country has made the fruits attractive for our study. The popularity of these fruits is due to their aromatic scent as well as their taste. According to the results of our study, the main components of the essential oils in the peels of these fruits (limonene and linalool) are in very high amounts in the essential oils and these fruits can be used as a source of these active substances. Therefore, the findings of our study are important. The aim of our study is to raise awareness about the value and usage areas of citrus peels, which are evaluated as waste. Elemental analysis of peels provides information about qualitative and quantitative analysis. Considering the aromatic structure of the peels, essential oil content becomes important. The results we obtained will add richness and innovation to the studies on this subject in the literature.

Material Method

The anhydrous sodium sulfate we used in our study was obtained from Merck.

Citrus material and isolation of essential oils

In this study the fruit of citrus (Lamas Lemon, Washington Navel Orange, Clementine Mandarin, Kumquat and Grapefruit) obtained from Antalya in Turkey in 2022. The fruits were used within the week they were harvested. The essential oils of the peels of the fruits were extracted with the help of the clevenger device. For this reason, fresh fruit peels were used in thick pieces. To isolate the essential oil 500 mg fresh peel added 3 litres of water and Hydro-distillation was performed for three h. The essential oils were dried over anhydrous sodium sulfate.

GC/FID-MS Analysis method

For identifying the components of the essential oil, a gas chromatography (GC) system (Agilent Technologies, 7890B) equipped with a flame ionization detector (FID) and coupled to a mass spectrometry (MSD) detector (Agilent Technologies, 5977A) was used. An HP-Innowax column (Agilent 19091N-116: 60 m×0.320 mm internal diameter and 0.25 um film thickness) was used to separate the compounds. The samples were analyzed with the column held initially at 70 °C after injection with 5 min hold time, then increased to 160 °C with a 3 °C min⁻¹ heating ramp. Finally, the temperature was raised to 250 °C with a 6 °C min-1 heating ramp with 5 min hold time. The carrier gas was helium (99.99% purity) with 1.3 mL min⁻¹ flow. The injection volume was set at 1 μ L (20 μ L essential oil was solved in 1 mL n-Hexane) with 8.20 minutes solvent delay. The injection was performed in split mode (40:1). Detector, injector, and ion source temperatures were 270 °C, 250 °C, and 250 °C, respectively. MS scan range was (m/z): 35-450 atomic mass units (AMU) under electron impact (EI) ionization of 70 Ev (Türk Baydir et al., 2021).

SEM-EDX analysis method

LEO 1430 VP model SEM device works with W (Tungsten) filament. There are secondary electron, backscattered electron and RÖNTEC QX2 brand and model XFlash type X-rays (EDX – Energy Dispersive X-ray Spectroscopy) detector on the device. The samples were carbon coated with the help of BAL-TEC SCD 005 Sputter coater. The ashes of the peels were used for Sem-edx analysis, images were taken from two different parts of the peels and analyzed, and the mean and standard deviation s values were calculated.

Determination of % dry matter and ash content Porcelain crucibles brought to constant weight were used for determination of % dry matter and ash. The % dry matter amount was made in an oven at 105°C and the amount of ash in a 500°C ash oven by keeping the fruits at least 6 hours.

Results

The results of the GC-MS were given in table 1.

To the Table 1, Limonene was detected as a common component in all fruit peels, from the lowest to the highest in order, approximately 24% in clementine mandarin, 28% in orange 76% in lemon, 98% in grapefruit 100% kumquat. The essential oils of clementine mandarin and orange peels were rich in linalool and measured 43 and 50%, respectively. About 22% α -terpineol component was detected in essential oils of orange peels. The Z-citral component was detected 13% in Clementine mandarin, while it was tested in lemon around 1%. Around 10% of γ -terpinene components were de-

tected in lemon peels(Table 1).

The dry matter ash matter and % essential oil of the peels were given in Table 2. To the Table 2 the % essential oil content of the peels was tested at maximum 7.20 in grapefruit and the lowest value of 2.1 was tested on kumquat fruit. % Dry matter was determined in crementin mandarin with the lowest value of 6.94. According to the results of ash determination in dry matter, it was tested in lemon with the lowest 2.88%. the mainly component of lemon grapefruit and kumquat were limonene. But minor and numeric quantities changes.

The essential oil component of mandarin(citrus reticulata) peels has been tested as limonene (75.16%), terpine-4-ol 10.36% (Abdel-Aziz et al., 2019). The chemical composition of the mechanically pressed essential oil of Nanfeng mandarin was tested as 56.76% limonene, 12.10% β -pinene, 12.03 γ -terpinene(Yi et al., 2018). According to the

Table 1: Essential oil composition of the peels of different citrus fruits

Tablo 1.farklı turunçgil me	yvelerini	n kabuklarır	ıın uçucu yağ ko	mpozisyonu	
Sample ID	РК	Rİ	RT	Area %	Component name
	1	1204	18,5718	24,4525	dl-Limonene
Mandarin clementine	2	1544	32,2301	43,4926	Linalool
	3	1390	38,2725	18,7916	3-Cyclohexene-1-methanol
	4	1707	39,9205	13,2634	Z-Citral
	1	1032	12,8841	0,708	α-Pinene
Grapefruit	2	1174	17,0783	1,735	β-Myrcene
-	3	1204	18,8006	97,557	dl-Limonene
	1	1032	12,884	1,9822	α-pinene
	2	1121	15,436	5,0823	β- pinene
	3	1125	15,7965	1,0993	Sabinene
	4	1174	17,0839	1,4956	β-Myrcene
Lamas Lemon	5	1204	18,7662	75,5734	dl-Limonene
	6	1047	20,3912	10,9852	γ- terpinen
	7	1707	37,8604	1,0249	Z-Citral
	8	1718	39,926	1,3771	2,6-Octadienal
	9	1758	42,3636	0,6192	2,6-Octadien-1-ol
	1	1204	18,5774	28,2045	dl-Limonene
Washington Navel Orange	2	1544	32,2357	50,1097	Linalool
	3	1701	38,2781	21,6859	α-terpineol
Kumquat	1	1204	18,56	100	dl-limonen

Table 2. % Dry matter ash matter and essential oil of the peels	
Cizelge 2 Kabukların kuru madde kül ve ucucu yağ yüzdeleri	

	% Dry matter	% Ash	% Essential oil
Kumquat	9.58±1.99	3.06	2.10
Grapefruit	9.18±1.05	3.40	7.20
Crementin mandarin	6.94±0.95	3.15	2.8
Lamas lemon	9.57±4.19	2.88	3.9
Orange	9.52±0.21	3.72	4.2

The ashes of the peels were used for Sem-edx analysis, images were taken from two different parts of the peels, and the mean and standard deviation s values were calculated (Figure 1, 2, 3, 4, 5 and Table 3).

Discussion and Conclusion

Citrus fruits can be consumed fresh or processed and used as additives in fruit juices, jams, carbonated beverages and food products. Citrus fruits, which are rich in vitamin C, have very important benefits for human health.

The main component of the essential oil of lemon peel grapefruit and kumquat peel was dl-limonen to the scientific researchs (Dao et al., 2021; X. Liu et al., 2019; Özogul et al., 2021). When the results of our study were evaluated, it was determined that results of our study, linalool was the dominant component in clementine mandarin.

Limonene (95.96%) and β -mircene(2.35%) were determined in the chemical composition of orange essential oil (Amaral et al., 2019). Yet another study on orange essential oil composition tested the major components as 96% limonene and 4% β -mircene (Radünz et al., 2021). In another study, linalool, geraniol and nerol, mainly limonene, are the other components detected in orange peel essential oil (Geraci et al., 2017). To the our study in orange peel eo the main component was linalool. The other components of orange eo were dl-limonene and α -terpineol. The difference of essential oil components is affected by variables such as light, the region where the plant grows, soil structure, plant type, genetic feature, age, harvest season

(Barra, 2009). This situation is compatible with the results of the literature and the results of our study (Galvan-Lima et al., 2021).

According to the results of our study, grapefruit and kumquat can be used as a source of limonene due to its limonene content, in line with literature studies (Maggiolino et al., 2022). mined as minor common components in all peels. Sodium and chlorine element were tested as minor common elements in kumquat and lemon peels. This should be taken into consideration in special uses.

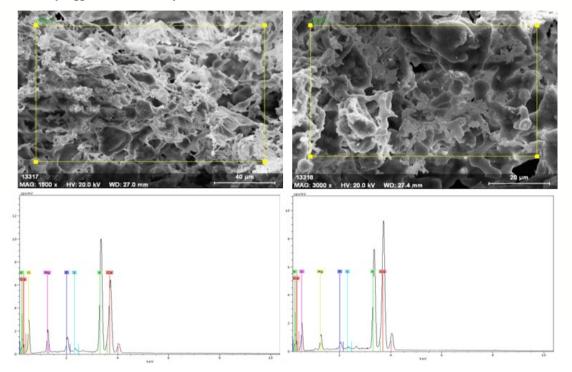


Figure 1. SEM image and edx chromatogram of grapefruit fruit peel Sekil 1. Greyfurt meyve kabuğunun SEM görüntüsü ve edx kromotogramı

Limonene is a monoterpene with antifungal action found in citrus essential oils (Ahmedi et al., 2022). Its antimicrobial (Han et al., 2020), antioxidant and anti-inflammatory (de Souza et al., 2019), anticancer (Chidambara Murthy et al., 2012) effects increase its value and importance. At the same time linalool components has antibacterial (He et al., 2022; Parasuraman et al., 2022) anti-cancer (Sun et al., 2015) affect. With the aid of atomic absorption spectroscopy, mineral matter analysis in dry ash samples was carried out in the peel and pulp of 4 different citrus samples. Eight minerals were identified, including 4 major elements (K, Ca, Na, and Mg) and 4 trace elements (Cu, Fe, Mn, and Zn) with the help of atomic absorption spectroscopy. Analysis was carried out by the dry ash samples. The most abundant element in peels was potassium, followed by calcium and magnesium, respectively (De Moraes Barros et al., 2012). According to the edx analysis results, grapefruit and orange peel are rich in calcium, while kumquat lemon and mandarin are rich in potassium. Mg, P and S were deterThis situation can also be benefited from in terms of soil in fruit growing. According to the results of the analysis, we can say that the peels are quite rich in terms of calcium and potassium (Figure 1, Table 3). Due to the rich content of the peels, it is recommended to add them to food products and to evaluate them in appropriate areas.



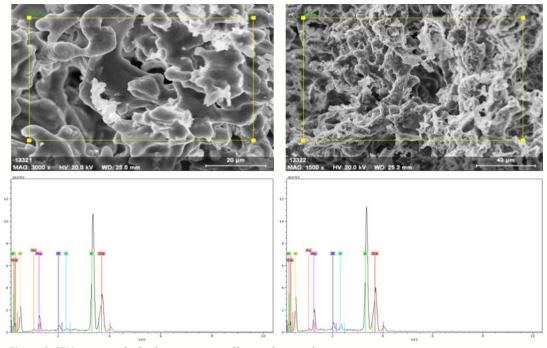


Figure 2. SEM image and edx chromatogram of lemon fruit peel Şekil 2. Limon meyve kabuğunun SEM görüntüsü ve edx kromotogramı

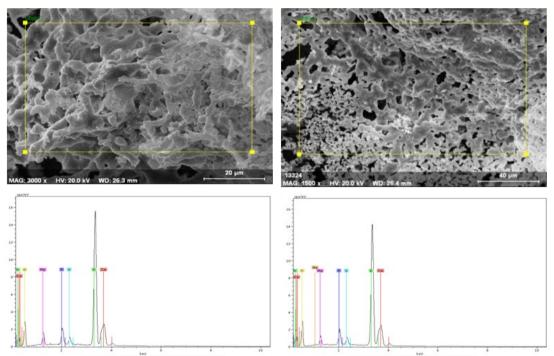


Figure 3. SEM image and edx chromatogram of mandarin fruit peel Şekil 3. Mandalina meyve kabuğunun SEM görüntüsü ve edx kromotogramı

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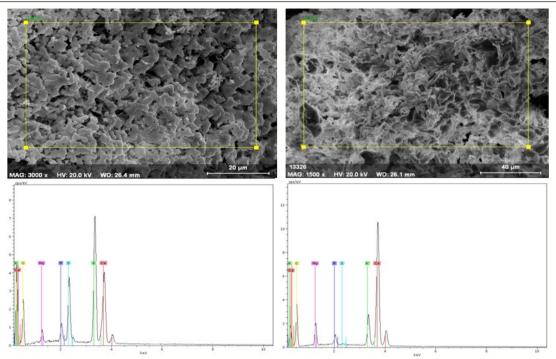


Figure 4. SEM image and edx chromatogram of orange fruit peel Şekil 4. Portakal meyve kabuğunun SEM görüntüsü ve edx kromotogramı

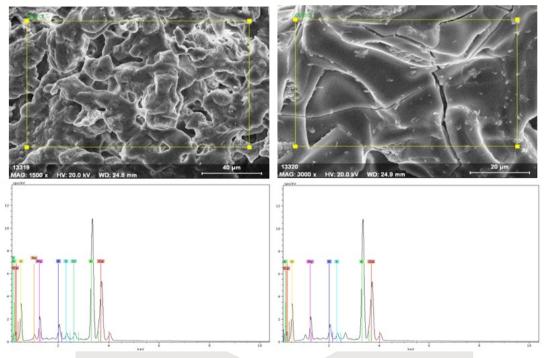


Figure 5. SEM image and edx chromatogram of kumquat fruit peel Şekil 5. Kumkuat meyve kabuğunun SEM görüntüsü ve edx kromotogramı

	Grapefruit	Kumquat	Lemon	Mandarin	Orange
Oxygen	42.49±3.10	42.51±0.30	43.91±0.75	47.12±0.38	51.96±3.82
Magnesium	3.51±1.36	5.05 ± 0.07	4.80±0.39	2.45±0.07	2.91±1.87
Phosphorus	1.73±1.14	2.76±0.04	1.56±0.19	3.26±0.19	2.01±0.30
Sulfur	0.55±0.27	1.22 ± 0.01	1.01±0.20	1.56±0.28	4±5.56
Potassium	22.58±3.44	26.26±0.03	32.15±1.33	35.89±0.94	13.68±11.26
Calcium	29.16±9.30	18.20±0.04	15.67±0.22	9.49±0.18	25.45±11.43
Sodium		2.14±0.01	0.93±0.01		
Chlorine		1.36±0.24	0.57±0.01		
Total	100	99.485	100.57	99.75	100.005

Table 3. SEM-EDX results of the peels (%	weight)
Table 3 Kabukların SFM edy sonucları	(% ağırlık

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