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Research Article

Evaluation of some physiochemical and antioxidant properties of some Syrian *Citrus* L. species

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Abstract: *Citrus* species have been cultivated throughout Syria as fruit trees, but the antioxidant properties of their fruits have not been fully revealed. The current study aimed to characterize the physiochemical, antioxidant compounds with their antioxidant activities of eight Syrian citrus juices (orange, sour orange, mandarin, lemon, citron, grapefruit, pomelo, and kumquat). The fruits were assayed for pH, titratable acidity, total soluble solids (TSS), ash, reducing sugar content, ascorbic acid, total phenolic compounds, total flavonoid content, carotenoids, and antioxidant activity. Of all Syrian *Citrus* species, the orange had the highest TSS content (11.8%) and the highest content of ascorbic acid (139.45 mg/100 g). Lemon was dominated by the high amount of carotenoids (77.56 mg β-carotene/100 g). Grapefruit demonstrated the highest content of total phenolic (659 mg GAE/100 g), total flavonoid compounds (326.49 mg quercetin/100 g), and antioxidant activity (87.94%). Pearson correlation analysis revealed that the phenolic compounds were the main contributing compounds.

INTRODUCTION

Citrus species fruits are members of the family *Rutaceae*. The genus *Citrus* consists of 203 species of evergreen shrubs or trees (Rivera *et al*., 2022). It is widely distributed all over the world, as its native occurs in Southeast Asia (mainly India and China) (Vashisth & Kadyampakeni, 2020), and cultivated varieties are grown in the Middle East, the Mediterranean, Europe, and the Americas. The *Citrus* trees are not frost-hardy (Wu *et al*., 2018). Thus, they are grown in a consistently sunny and humid environment. The *Citrus* fruit's colour ranges between yellowish green, yellow/orange/red when mature. Fruits are usually of a large size, with a subacidic to sweet taste.

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Besides orange (*Citrus sinensis* (L.) Osbeck), sour orange or also known as bitter orange (*Citrus aurantium* L.), mandarin (*Citrus reticulata* Blanco), and lemon (*Citrus limon* (L.) Osbeck) many Syrian fruits are also extensively used as citron (*Citrus medica* L.), grapefruit (*Citrus paradisi* Macfad.), shaddock, also known as a pomelo (*Citrus maxima* (Burm.) Merr.), as food ingredients and therapeutic agents for health. On the other hand, kumquat (*Citrus japonica* Thunb.) is rarely used, thus grown in limited areas of Syria.

Citrus species are consumed increasingly in fresh and also used in the preparation of jams and beverages, due to its distinctive aroma, taste and flavor (Al-Juhaimi & Ghafoor, 2013), as well as their phytonutrients contain a natural sources of bioactive substances (Pellegrini *et al*., 2003; Peterson *et al*., 2006), which associated with reduced of chronic diseases risk, such as asthma (Dugo & Giacomo, 2002) by its have many antioxidant, anti-inflammatory, anti-fungal, antimicrobial, antiviral, anti-tumor and blood clot inhibition properties (Abeysinghe *et al*., 2007; Cerda *et al*., 1994; Chiu & Chang, 1998; Garg *et al*., 2001; Kaur & Kapoor, 2001). These bioactive substances, which are responsible for the prevention of degenerative disease, include ascorbic acid, carotenoids (Alquézar *et al*., 2008), and phenolic compounds (Al-Juhaimi & Ghafoor, 2013). Ascorbic acid is another water-soluble vitamin, which is found in the flesh and peel of citrus fruits. The content of ascorbic acid in pomelo (*C. maxima*), orange (*C. sinensis*) can reach 60 mg/100 g and 30 mg/100 g, respectively (Ye, 2005). Carotenoids are a group of tetraterpenoids, which can quench $^1O2$ and eliminate harmful free radicals (Di Mascio *et al*., 1989) to show their antioxidant properties. These phenolic compounds are categorized as phenolic acids and flavonoids (Balasundram *et al*., 2006). Phenolic compounds, which comprise a group of bioactive compounds that include flavonoids (Huet, 1982; Zou *et al*., 2016), are the most studied in the previous literature. Flavonoids have antioxidant properties with an excellent hydroxyl radical scavenging activity (Geroulakos & Nicolaides, 1994; Loizzo *et al*., 2012).

However, no reliable reports on the phytochemical composition of different *Citrus* species in Syria exist, further emphasizing the lack of knowledge on the chemical properties that reveal its therapeutic potential benefits.

In this study, sour orange, orange, mandarin, lemon, citron, grapefruit, pomelo and kumquat samples were collected from central Damascus market in Syria, and then subjected to physiochemical properties such as total soluble solids, titratable acidity (TA), pH and total soluble solids/titratable acidity (TSS/TA) ratio to detect quality of the *Citrus* fruits. Carotenoids, ascorbic acid, polyphenolic, and flavonoid compounds with their antioxidant activity were analysed using a spectrophotometer. Therefore, the obtained results could contribute to our understanding of the phytochemical and antioxidant properties of different *Citrus* spp. in Syria.

MATERIAL and METHODS

Collection of *Citrus* Species Samples

During 2022, fully matured *Citrus* species (*C. sinensis*, *C. aurantium*, *C. reticulata*, *C. limon*, *C. medica*, *C. paradisi*, *C. maxima,* and *C. japonica*) fruits were randomly taken from the central Damascus market in Syria. Samples were brought to the Food and Industrial Biotechnology Laboratory, National Commission for Biotechnology (NCBT), Damascus, Syria. Fruits were squeezed under room conditions. After the squeezing processes, the juices were stored at -20 ^{o}C until further testing.

Physiochemical Characteristics

Total soluble solids

Each sample was analysed for TSS using a refractometer (KRÜSS, Germany).

Titratable acidity

TA was determined as citric acid percentage according to AOAC (2000).

pH

The pH was measured using a pH meter (pH meter, Model: Precisa 900, Switzerland). First, the pH meter was standardized using buffer solutions of pH 4.0 and 7.0. Then, the pH of each *Citrus* species was measured. The pH probe was rinsed using distilled water after each measurement.

Total soluble solids/Titratable acidity

TSS/TA was calculated by dividing the TSS of each *Citrus* species juice by its TA according to the previously described method by Grewal *et al*. (2000) and Lacey *et al*. (2009).

Ash

The ash was measured as a percentage according to the method described by AOAC (2000). Accordingly, about 5 g of each sample was burned at 550°C for 5 h in an ash oven, then its residue was weighed to calculate the ash content of each sample as g per 100 g of juice using the following equation:

$$\text{Ash content} = W1 - W2$$

W1: is the weight of ash in the crucible

W2: is the weight of an empty crucible.

Total reducing sugar

The total reducing sugar (as g/100 g) of *Citrus* juices was determined as described previously by AOAC (2000).

Antioxidant Properties

Ascorbic acid

The quantification of ascorbic acid in *Citrus* juice was done using 2,6-dichlorophenolindophenol (DCPIP), which is generally used as an indicator to express ascorbic acid content as mg/100 g (AOAC, 2000). Approximately 5 g of each juice was mixed immediately with 1 mL glacial acetic acid before titration to maintain the acidic environment ($pH \approx 4$) to ensure the optimal stability of ascorbic acid, facilitate the complete dye reduction, and ensure the quantitative reaction (Ojukwu & Nwobi, 2017; Ruiz *et al*., 2018). Then, the standard dye solution was incrementally added to each sample until a light pink color persisted, referring to the endpoint of titration. The burette reading was measured to determine the volumes of the consumed standard solution. The titration was carried out in triplicate for each sample, and the volumes of consumed standard solution were measured for each replicate.

Total carotenoid

The analysis of carotenoids was performed by absorbance measurement in the spectrophotometer (OPTIZEN, Korea) as described previously in the published method by Lee and Castle (2001). Approximately 1 g of each sample was homogenized by breaking into 10 mL of hexane, acetone, ethanol (50: 25: 25, v: v: v) and then centrifuged at 6500 rpm for 5 min. The supernatant was immediately added to a 25 mL volumetric flask, and the total volume was made up to 25 mL with hexane. Then it was absorbance at 450 nm using the spectrophotometer. The amount of carotenoids was calculated as milligrams of β-carotene per 100 grams of sample (mg β-carotene/100 g).

Total phenolic compounds

The phenolic compounds were extracted by modifying the Wada & Ou (2002) method as follows. Briefly, 1 g of the sample was mixed with 30 mL of methanol for 15 min at room temperature on a magnetic stirrer operated at high speed and then centrifuged at 3000 rpm, and the supernatant was immediately analysed.

The quantification of phenolic compounds was evaluated by modifying the Folin-Ciocalteu assay (Asami *et al*., 2003) as follows. Approximately 3 mL of distilled water, 2 mL of sample, and 0.2 mL of Folin-Ciocalteu reagent were added to a 10 mL volumetric flask. The contents were mixed for 2 min at room temperature. Next, 4 mL of a 7% Na_2CO_3 was added, followed by the addition of distilled water to the volume. Then, it was mixed and allowed to stand for 2 h at room temperature. Next, they were filtered to determine the total phenol concentration using a spectrophotometer (OPTIZEN, Korea) at 750 nm. Total phenolic content was quantified as milligrams gallic acid equivalents per 100 g of sample (mg GAE/100 g).

Total flavonoids

Total flavonoids were prepared as the following steps. Approximately 1 g samples were extracted with 50 mL methanol (80%). Then, the extractions were performed on a magnetic stirrer operated at room temperature for 10 min.

Flavonoids were estimated by the $AlCl_3$ colorimetric method (Marinova *et al*., 2005). Approximately 1 mL of extraction with 4 mL dd H_2O was added to a 10 mL volumetric flask. Next, 0.3 mL of 5% $NaNO_2$ was added to the volumetric flask. After 5 min, 0.3 mL of 10% $AlCl_3$ was added. At the 6th min, 2 mL of NaOH (1 M) was added, and ddH2O was filled to volume. Then, the total flavonoids were measured using a spectrophotometer (OPTIZEN, Korea) at 510 nm. Qualitative data were presented as milligrams of quercetin per 100 grams of sample (mg quercetin/100 g).

Determination of antioxidant activity using the DPPH method

The 2, 2-diphenyl-1-picrylhydrazyl (DPPH) antioxidant activity assay was carried out using the procedure of Singh *et al*. (2002). DPPH solution (60 µmol/L methanol) was mixed with an equal volume of each *Citrus* extract (1 g of the sample with 100 mL methanol). After 30 min, the absorbance of the reaction mixture was measured at 517 nm. The following equation was utilized to calculate the DPPH of each extract:

$$DPPH\% = [(A - A')/A] \times 100$$

A: A control absorbance

A': A sample absorbance

Statistical Analysis

The data obtained was statistically analysed using SPSS (Version 17). The coefficient of determination (R) and the calibration curve were obtained using MS Excel 2019 (Microsoft, Redmond, WA, USA). The analytical measurements were performed in triplicate, and the results represent the means of the triplicates (for each measurement) with standard deviation (mean±SD). One-way ANOVA was used to determine if significant differences between means were found or not at $p<0.05$. It included a post hoc test (LSD).

RESULTS

Physicochemical Characteristics

TSS, TA, and pH

Different physicochemical characteristics, such as TA, TSS, and pH were presented in Table 1 were chosen to characterize different *Citrus* juices in terms of overall quality.

TSS was an important quality attribute of juices, and was higher in the orange (11.8%), however, the lowest TSS value (7.8%) was from the kumquat.

TA ranged between (0.72-6.64%), which was higher in the case of citron juice (6.64%), however, the mandarin juice had the lowest (0.72%) values.

The pH values of the different Syrian *Citrus* juices ranged between 2.65-3.73). Mandarin dominated the highest pH value (3.73) with the lowest acidity (0.72%). However, *Citrus medica* had the lowest pH value (2.65) with the highest acidity (6.64%).

The TSS/TA ratio of different *Citrus* juices was presented in Figure 1.

Table 1. Total soluble solids, titrable acidity and pH of different Syrian *Citrus* species.

Name	Scientific name	TSS (%)	TA (as citric acid%)	pH
Grapefruit	*Citrus paradisi*	10.35+0.04^{c}	1.46+0.02^{c}	3.03+0.01^{d}
Orange	*Citrus sinensis*	11.8+0.01^{d}	0.75+0.03^{a}	3.7+0.03^{e}
Lemon	*Citrus limon*	8.35+0.02^{b}	3.55+0.02^{d}	2.66+0.01^{a}
Pomelo	*Citrus maxima*	10.6+0.03^{c}	1.15+0.02^{b}	3.45+0.02^{c}
Sour orange	*Citrus aurantium*	10.5+0.02^{c}	4.21+0.03^{f}	2.91+0.02^{b}
Citron	*Citrus medica*	9.9+0.05^{c}	6.64+0.02^{g}	2.65+0.01^{a}
Mandarin	*Citrus reticulata*	10.4+0.03^{c}	0.72+0.03^{a}	3.73+0.03^{e}
Kumquat	*Citrus japonica*	7.8+0.07^{a}	4+0.01^{e}	2.87+0.03^{b}

The different letters indicate significant differences within the same column. LSD test: For TSS (SE=0.05286), TA (SE=0.02708), pH (SE=0.02466).

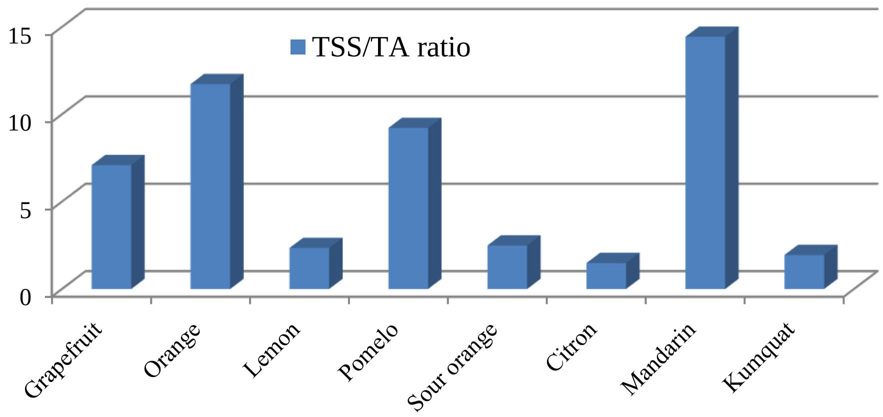

Figure 1. TSS/TA ratio of different Syrian *Citrus* species.

TSS/TA ratio of Syrian *Citrus* fruits ranged between (1.49-14.44). Mandarin had the highest TSS/TA ratio, whereas citron had the lowest (1.49).

Ash and reducing sugar

Ash with reducing sugar in different Syrian *Citrus* fruits is presented in Table 2. Ash in different Syrian *Citrus* species ranged between (0.17-0.44%). The ash of *C. paradisi*, *C. sinensis*, *C. maxima*, *C. medica*, *C. reticulata,* and *C. japonica* juices did not differ significantly (Table 2), but that of *C. aurantium* juice was revealed as the highest ash content (0.44), and that *C. limon* had the lowest content (0.17). Reducing sugar content in Syrian orange fruits was high (4.46 g/100 g). The lowest reducing sugar content (2.15 g/100 g) was found in citron fruits.

Table 2. Ash with reducing sugar of different Syrian *Citrus* species.

Name	Scientific name	Ash (%)	Reducing sugar(as g/100 g)
Grapefruit	*Citrus paradisi*	0.36+0.03^{b}	3.85+0.04^{d}
Orange	*Citrus sinensis*	0.34+0.02^{b}	4.46+0.09^{e}
Lemon	*Citrus limon*	0.17+0.01^{a}	2.97+0.08^{b}
Pomelo	*Citrus maxima*	0.38+0.02^{b}	3.26+0.07^{c}
Sour orange	*Citrus aurantium*	0.44+0.01^{c}	3.18+0.05^{c}
Citron	*Citrus medica*	0.33+0.02^{b}	2.15+0.06^{a}
Mandarin	*Citrus reticulata*	0.37+0.02^{b}	2.84+0.07^{b}

Kumquat	*Citrus japonica*	0.25+0.02[b]	3.7+0.02[d]

The different letters indicate significant differences within the same column. LSD test: For Ash (SE=0.01472), and for reducing sugar (SE=0.04836).

Antioxidant Properties

Ascorbic acid and carotenoid

Ascorbic acid in eight different Syrian c *Citrus* juices is presented in Table 3. The ascorbic acid contents of grapefruit, citron, lemon, pomelo, and mandarin juices did not differ significantly, but those of orange juice were markedly higher (139.45 mg/100 g), and the sour orange had the lowest ascorbic acid content (mg/100 g).

Table 3. Ascorbic acid and carotenoid in different Syrian *Citrus* species.

Name	Scientific name	Ascorbic acid (mg/100 g)	Carotenoids (mg β-carotene/100 g)
Grapefruit	*Citrus paradisi*	108.56+0.81[b]	73.28+3.68[e]
Orange	*Citrus sinensis*	139.45+0.38[c]	37.78+2.4[d]
Lemon	*Citrus limon*	106.32+1.16[b]	77.56+3.08[e]
Pomelo	*Citrus maxima*	106.32+0.53[b]	25.74+2.34[c]
Sour orange	*Citrus aurantium*	99.05+0.62[a]	12.25+1.9[b]
Citron	*Citrus medica*	106.63+0.2[b]	14.66+1.83[b]
Mandarin	*Citrus reticulata*	105.37+0.99[b]	16.72+2.14[b]
Kumquat	*Citrus japonica*	104.22+0.75[b]	7.3+0.85[a]

The different letters indicate significant differences within the same column. LSD test: For ascorbic acid (SE=0.55381) and for carotenoids (SE=1.82769).

The results from this study on the quantity of carotenoids found in different Syrian *Citrus* species are presented in Table 3. Total carotenoid was dominant in lemon fruits (77.56 mg β-carotene/100g). However, the lowest carotenoids were found in Kumquat (7.3 mg β-carotene/100g).

Total phenolic and flavonoid compounds

The total phenolic compounds in Syrian *Citrus* fruits are presented in Table 4. Grapefruit juice was the best in terms of phenolic compounds, being the maximum (659 mg GAE/100 g), followed by mandarin juice (613 mg GAE/100 g). The Kumquat had the lowest amount of these bioactive compounds (295.6 mg GAE/100 g).

Table 4. Total phenolic and flavonoid compounds in different Syrian C *Citrus* species.

Name	Scientific name	Phenolic compounds (mg GAE/100 g)	Flavonoid compounds (mg quercetin/100 g)
Grapefruit	*Citrus paradisi*	659.015+4.12[h]	326.49+7.62[f]
Orange	*Citrus sinensis*	329.18+8.22[b]	94.42+3.16[a]
Lemon	*Citrus limon*	466.43+5.38[d]	121.4+4.12[b]
Pomelo	*Citrus maxima*	432.48+4.18[c]	206.7+5.88[e]
Sour orange	*Citrus aurantium*	534.68+2.55[f]	131.09+2.35[c]
Citron	*Citrus medica*	496.68+2.87[e]	104.98+2.03[a]
Mandarin	*Citrus reticulata*	613.13+3.68[g]	147.88+3.46[d]
Kumquat	*Citrus japonica*	295.6+5.32[a]	101.84+6.75[a]

The different letters indicate significant differences within the same column. LSD test: For phenolic compounds (SE=3.95669), and for flavonoid compounds (SE=3.92498).

The level of total flavonoids in Syrian *Citrus* s fruits was reached (326.49 mg quercetin/100 g) in grapefruit juice (Table 4). The low level of flavonoids was found in orange (94.42 mg quercetin/100 g).

Antioxidant activity

In the present study, the antioxidant activity of Syrian *Citrus* fruits was evaluated by the DPPH method (Table 5). The antioxidant potential of *Citrus* fruits was in the range of 48.92 - 87.94 %. Grapefruit demonstrated the highest antioxidant activity (87.94%), while the lowest antioxidant activity was in the kumquat.

Table 5. Antioxidant activity using DPPH method in different Syrian Citrus species.

Name	Scientific name	Antioxidant activity (%)
Grapefruit	*Citrus paradisi*	87.94+3.97^{d}
Orange	*Citrus sinensis*	64.635+2.28^{b}
Lemon	*Citrus limon*	49.7+2.14^{a}
Pomelo	*Citrus maxima*	60.295+3.17^{b}
Sour orange	*Citrus aurantium*	75.2+4.11^{c}
Citron	*Citrus medica*	79+1.08^{c}
Mandarin	*Citrus reticulata*	84.82+1.38^{d}
Kumquat	*Citrus japonica*	48.92+1.85^{a}

The different letters indicate significant differences within the same column. LSD test: For antioxidant activity (SE=2.35200).

Correlation analysis

The correlation coefficients (R) of ascorbic acid, carotenoids, phenolic, and flavonoid compounds with their antioxidant activity of eight different Syrian *Citrus* species are presented in Table 6, 7, 8, 9 and Figure 2.

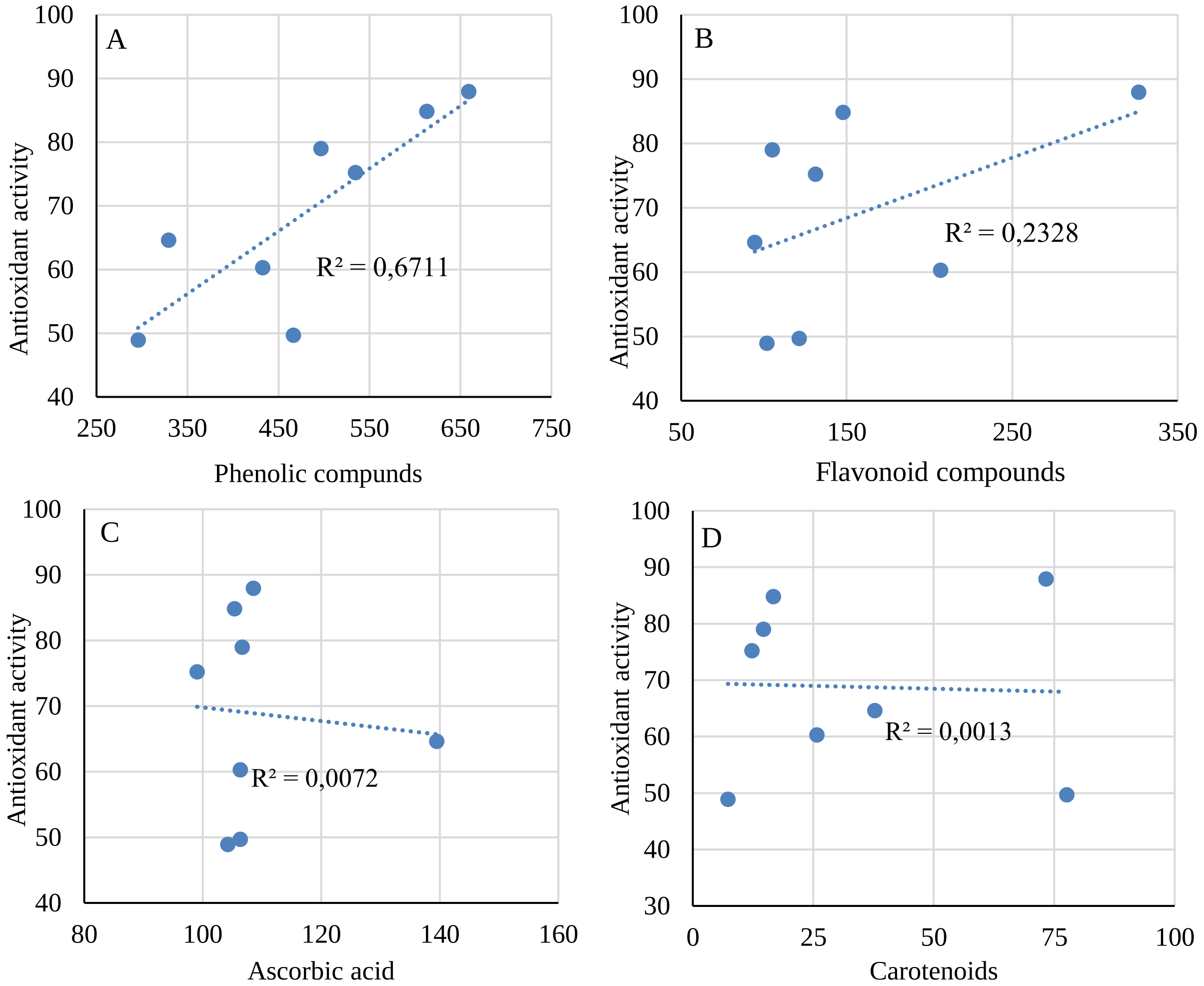

Figure 2. Correlation analysis of ascorbic acid, carotenoids, phenolic, and flavonoid compounds with their antioxidant activity using DPPH method in different Syrian *Citrus* species.

Pearson correlation analysis of phenolic compounds with DPPH (Figure 2A) was 0.819 (p=0.013, p < 0.05), indicating a good positive relationship. While the correlation analysis between flavonoids and DPPH (Figure 2B) showed a correlation coefficient of 0.483 (p=0.226, p > 0.05), no clear positive relationship was observed. However, the correlation coefficients of ascorbic acid and carotenoids with DPPH were -0.085 (p=0.931, p > 0.05) (Figure 2C), -0.037 (p=0.931, p > 0.05) (Figure 2D), respectively, revealing no relationship could be found.

DISCUSSION

Among different quality parameters of all eight *Citrus* species grown in Syria, *C. sinensis* demonstrated the highest TSS (11.8%), whereas *C. japonica* had the lowest content (7.8%). The highest TSS in orange reflects its high quality, which was in agreement with the findings of Al-Juhaimi and Ghafoor (2013) in which a similar trend in the content of TSS was observed in the lemon and orange juices. However, our results differ from that report because therein, the quantity of TSS of mandarin juice was shown to be higher than TSS of orange juice. Our TSS results in grapefruit were also consistent with Ahmed *et al.* (2019) in that low contents of TSS were recorded in 8 Indian grapefruit varieties. Moreover, our results were also consistent with some other reports of Sicari *et al*. (2018), in which a similar content of TSS in two varieties of grapefruits. These total soluble solids increase with the ripening of *Citrus* fruits as well as their high content of sugars (Izquierdo & Sendra, 2003; Al-Juhaimi & Ghafoor, 2013).

Titratable acidity was the highest content in *C. medica*, while the lowest content was in *C. reticulata*. The Syrian mandarin juice was observed to be less acidic, reflecting its considerable sweetness. This TA was lower than the finding of Al-Juhaimi and Ghafoor (2013) in mandarin grown in Saudi Arabia.

The pH values of the different Syrian *Citrus* juices ranged between 2.65-3.73). These results are influenced by the stages of the fruit ripening with their maturity (Falade *et al*., 2003). These findings align with previous reports on various *Citrus* species (Al-Juhaimi & Ghafoor, 2013; Sicair *et al*., 2018; Ahmed *et al*., 2019). The decrease in the pH value refers to the more acidic *Citrus* juice (Tiencheu *et al*., 2021). The pH value in Syrian grapefruit was lower than the findings of Ahmed *et al*. (2019) in different Indian grapefruit varieties and it was in accordance with reported values for two Italian cultivated varieties (Sicair *et al*., 2018). Also, the pH value of Syrian lemon, orange, and mandarin fruits was higher than the findings of Al-Juhaimi and Ghafoor (2013) in that lemon, orange, and mandarin juices were grown in Saudi Arabia.

TSS/TA ratio of the different Syrian *Citrus* species ranged between (1.49-14.44). The highest TSS/TA ratio in *C. reticulata* reflects its high quality, which increases with the increase of ripening of the *Citrus* fruits as well as their high content of sugars (Izquierdo & Sendra, 2003; Al-Juhaimi & Ghafoor, 2013).

The ash in different Syrian *Citrus* juices ranged 0.17% in *C. limon* and 0.44% in *C. aurantium*. Ash in grapefruit, sour orange, and mandarin fruits grown in Syria were close to those values reported by Sidana *et al*. (2013). However, our results were lower than those reported in the orange and lemon fruits. These ashes could be related to the presence of microminerals and macrominerals in *Citrus* juices, which can play a crucial role in the health benefits (Dawadi *et al*., 2022; Ruperez, 2002) by influencing nerve and muscle function, creating the structural components of the bones, and regulating the water balance in the body (Kim & Choi, 2013).

Ascorbic acid in Syrian *Citrus* fruits was higher than the findings of Sidana *et al*. (2013) in the mandarin, orange, and lemon fruits. This ascorbic acid of Syrian lemon was also higher than the report of Al-Juhaimi and Ghafoor (2013) in that lemon grown in Saudi Arabia. However, Ascorbic acid in Syrian grapefruit was lower reported in two different grapefruit varieties (Sicair *et al*., 2018). These differences might be due to analytical differences (Al-Juhaimi & Ghafoor, 2013), *Citrus* species effects, the difference in climate and region (Isbilir *et al*., 2012), cultural practices, fruit quality, maturity, packaging, and storage conditions (Martí *et al*., 2009).

Carotenoids are natural coloring substances widely distributed in vegetal raw materials. They find enormous application not only as colorants, but also as bioactive substances in food fortification as provitamin A. Our data were higher than those findings of Alquézar *et al*. (2008) in grapefruit and lemon.

Total phenolic compounds of *Citrus* juices ranged from 295.6 mg GAE/100 g in *C. japonica* to 659 mg GAE/100 g in *C. paradisi*. The polyphenolic compounds in the Syrian grapefruit were higher than the reported quantity of polyphenols for two varieties of *C. paradisi* (Sicair *et al*., 2018). Moreover, our report recorded higher polyphenolic compounds than those found in orange, mandarin, and lemon fruits (Al-Juhaimi & Ghafoor, 2013). Variations in extraction methods, solvents employed, and the type of sample might account for these differences (Fejzić & Ćavar, 2014).

The total flavonoid compounds ranged from 94.42 mg quercetin/100 g in *C. sinensis* to 326.49 mg quercetin/100 g in *C. paradisi*. These findings were consistent with the study of Sicari *et al*. (2018) on the flavonoids in two grapefruit (Marsh & Star Ruby) varieties. Differentiation of flavonoids in different Syrian *Citrus* species could be due to many factors, such as cultivation and harvesting practices, ripeness, environmental conditions, and genetic makeup (Sharma *et al*., 2017).

Among these *Citrus* juices, grapefruit juice has the highest DPPH values (87.94%). The differences in antioxidant activity using DPPH method for different Syrian *Citrus* species could be related to the phenolic compounds (Franke *et al*., 2004) in *Citrus* fruits, which are highly bioavailable antioxidants, effectively scavenging reactive oxygen species (Zou *et al*., 2016; Nakao *et al*., 2011) and the *Citrus* species with a higher phenolic compounds show a stronger antioxidant *activity. There are different factors that may contribute to these differential results, such as the Citrus* species, stage of maturity, and the methodology of the antioxidant analyses. Syrian grapefruit has high antioxidant activity compared with the antioxidant activity of eight different Indian grapefruit varieties (Ahmed *et al*., 2019) and two different Italian grapefruit varieties (Sicair *et al*., 2018). This high antioxidant activity of Syrian grapefruit could be explained by the higher number of –OH groups, and those –OH groups present in ortho-position in the aromatic ring usually quench more DPPH molecules on a molar basis (Dadwal *et al*., 2018). This revealed a good correlation between total polyphenol compounds and their DPPH antioxidant activities, which were statistically significant in Syrian grapefruits. Therefore, *C. paradise* is a rich source of valuable compounds that could be utilized in foods and health.

The correlation coefficients of phenolic compounds with DPPH were 0.6711 ($p < 0.05$), indicating a good relationship. Accordingly, the phenolic compounds were a highly contributing component in the higher antioxidant activity of Syrian *Citrus* fruits (Chen *et al*., 2010; Hua *et al*., 2018). Moreover, Hua *et al*. (2018) found a good correlation between phenolic compounds of Chinese *Citrus* juices and their antioxidant activity using the DPPH method. Although the good relationship between the phenolic compounds with their antioxidant activity, the phenolic compounds were not the sole contributing component, allowing other substances to contribute to antioxidant activity in these Syrian *Citrus* juices. Another possible reason was that more content of segment membranes and pulp into the Syrian *Citrus* juices due to the squeezing process, allowing other substances such as phenolic acids, coumarins, mineral elements, other vitamins (vitamin A, vitamin E, vitamin B1, B2, and B3), and pectin (Zhou, 2012) to play an antioxidant activity contributing role.

The correlation analysis between flavonoids and DPPH was 0.2328 ($p < 0.0.05$), revealing a good positive relationship could not be found. Although the flavonoids are the main component in the phenolic compounds of *Citrus* fruits. This result indicated that the flavonoids and DPPH relationship might reveal that flavonoids contribute less than phenolic compounds, according with the previously studied results that flavonoids play a less substantial antioxidant activity contributing role (Saeed *et al*., 2012; Li *et al*., 2018). However, Chen *et al*. (2010) noticed that the antioxidant activities of the *Citrus* flesh were highly correlated with the flavonoid compounds, indicating that these flavonoids act as a highly contributing component in the antioxidant activity of the *Citrus* juices (Bellocco *et al*., 2009).

The correlation coefficients of ascorbic acid and carotenoids with DPPH were 0.0072 (p < 0.05), 0.0013 (p < 0.05), respectively, revealing no relationship could be found. These findings were consistent with Tounsi *et al*. (2011), who found a negative relationship between β-carotene of Tunisian *Citrus* juices with their antioxidant activity.

CONCLUSION

This study demonstrated that the physicochemical parameters, antioxidant compounds, and antioxidant activity of *Citrus* juice from eight different Syrian *Citrus* species varied significantly. Grapefruit demonstrated the highest antioxidant activity with the highest content of its phenolic and flavonoid compounds. While lemon had the highest content of carotenoids. However, orange was reported markedly the highest content of ascorbic acid and TSS. These antioxidant compounds were detected in *Citrus* juices, which have known health benefits, as well as their role in the lipid peroxidation system. Our results will provide important information about the good quality and a highly source of ascorbic acid, carotenoids, phenolic, and flavonoid compounds with significant antioxidant potential in these *Citrus* species can play a crucial role in human health.

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Declaration of Conflicting Interests and Ethics

The authors declare no conflict of interest. This research study complies with research and publishing ethics. The scientific and legal responsibility for manuscripts published in IJSM belongs to the authors.

Data Availability Statement

Research data is available and can be sent with e-mail upon request.

Authorship Contribution Statement

Hala Khaled: Investigation, Methodology, Resources, Visualization, Formal Analysis, Software, and Writing-original draft. **Bassam Aloklah:** Project administration, Investigation, Methodology, Validation, and Supervision. **Mayadah A. Haj Ali:** Investigation, Methodology, Conceptualization, and Writing – review.

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