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Bioactive Compounds of Juice and Peels of Yuzu Fruits Cultivated in Switzerland

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Abstract: Yuzu is a citrus fruit cultivated mainly in northeast Asia. Due to the huge gastronomic interest emerging in recent years, some attempts to grow yuzu in Europe are being made. Juice and peel of yuzu cultivated in Switzerland have been characterised in this study. Peel constituted the major part of yuzu fruit followed by flesh, juice and seeds. The fruit degree of maturity was investigated by measuring pH, total titratable acidity and total soluble solids of the juice. The analyses were pursued by determining the content of vitamin C, malic and citric acids amounting to 0.560, 6.18 and 44.7 g/L, respectively. Four flavanones: naringin, hesperidin, naringenin, and hesperetin were identified and quantified in both juice and peel. The most abundant flavanone was hesperidin with 47 µg/mL and 640 µg/g dry matter (DM) in juice and peel, respectively. For the first time yuzu grown in Switzerland have been analysed and the obtained values have been compared with literature for other citrus fruits. Yuzu juice showed higher contents of citric and malic acid. Yuzu cultivated in Switzerland contained nearly twice as much vitamin C as yuzu juices from different regions of Japan. The content of vitamin C of yuzu was as high or higher than in most other citrus fruits. Large differences in the content of flavanones in yuzu juice from different regions of Japan were reported, in general the values noted were lower than those in the present study.

Keywords: Antioxidant activity · Flavanone · Juice · Peel · Vitamin C · Yuzu

1. Introduction

Citrus junos Siebold ex Tanaka better known as yuzu or yuja is a citrus fruit, belonging to the *Rutaceae* family. Native to East Asia, more precisely to China, it then spread to Korea and Japan. It is reported that yuzu is a natural hybrid between *Citrus ichangensis* (papeda) and *Citrus reticulata v. austera* (sour mandarin).[1] Its annual production in Japan in 2004 reached about 20 000 tons.[2] Yuzu trees are cold-hardy and very thorny. Fruits look like small grapefruits with diameter ranging from 5.5 to 7.5 cm (Fig. 1). They gained in popularity worldwide due to their pleasant flavour. Yuzu fruits are used to make vinegar, condiment for salad seasoning, sauces or marmalade, mainly in the gourmet restaurants. Fruit components are also used in cosmetics and perfumes manufacturing and more recently in aromatherapy.[2–4]

Apart from being recognised for their interesting flavour, yuzu fruits are becoming the subject of research worldwide owing to their content of bioactive substances. All constitutents of yuzu fruits *i.e.* juice, peel and seeds contain bioactives with diverse biological activities such as antioxidant, anti-inflammatory, antitumoral, antifungal or antiviral.[5–7] Furthermore, yuzu fruits were reported to play a role in preventing obesity or cardiovascular disorders.[7,8]

Fig. 1. Yuzu fruit.

Similarly to other citrus fruits, yuzu might be a source of vitamin C as well as phenolic compounds, mainly flavanones.[9,10] Juice, the most commonly consumed part, is rich in essential nutrients whereas polyphenols are present only in small quantities. By discarding peel, seeds and pomace, the natural phytochemical potential that they could provide is wasted. For this reason, it is of great interest to strengthen the knowledge on bioactive compounds of different parts of yuzu. It might encourage utilisation of peel in cuisine *i.e.* for yuzu zest sprinkled dishes for its nutraceutical application. Peel extract supplement may be effective for controlling fasting blood glucose levels and may serve as a useful supplement in subjects with prediabetes.[11]

Even if yuzu trees show the strongest cold resistance in citrus family, and it can grow in locations where winter temperature can drop down to –9 °C,[12] studies on yuzu cultivated in Europe are rather rare. The aim of this study was to identify bioactive compounds and their antioxidant activity in juice and peel of yuzu cultivated in Switzerland and to compare with literature values obtained for other citrus fruits.

2. Experimental

Additional experimental data is available in the supplementary material associated with this article on Chimia at *Ingentaconnect.com.*

2.1 *Plant Material*

Yuzu trees, a natural hybrid of *Citrus ichangensis* × *C. sunki*, called ichandarin, were cultivated in Borex (Switzerland). The trees were planted in the ground under a garden greenhouse with full sun exposure and heating possibility. The fruits were harvested in October 2015 at optimal ripeness and undamaged fruits were frozen immediately at –20 °C until analysis. For thawing, fruits were placed at 8 °C for 16 h. Fruits were randomly assigned to four different groups. Three fruits were pooled in a group, and groups were analysed separately. The fruits were cut in half and pressed manually using a juicer. The juice of the three fruits was put together, and both the weight and the volume were measured. Peel was separated from the pulp and it comprised of both flavedo and albedo. Seeds were removed manually from the pulp and washed with distilled water. If not analysed immediately, the separated parts of yuzu fruits were stored at –20 °C.

3. Results and Discussion

Different morphological parts of yuzu were separated and analysed. When possible, the obtained results were discussed with literature data on yuzu fruits. Additionally, the comparison between various citrus fruits has been made.

The yield of individual constituents was calculated (Table 1). Juice accounted for 17.3% of whole yuzu fruit. Juice yield was considerably different from that reported for yuzu originating from different regions in Japan ranging from 8.1 to 15.8%.[19] Much higher juice yields were obtained for other citrus fruits amounting to 27.0–60.7%.[20,21] It is comprehensible since yuzu fruits have roughly the same size as lemons or mandarins but their seeds are bigger and found in larger quantities. Peel of yuzu comprised both flavedo and albedo. The content of peel accounted for 44.2% of the whole fruit conforming to the value reported by Assefa & Keum.[22] Compared to other citrus fruits, which are composed of 50–65% of peel,[23] yuzus have slightly lower values.

Peel moisture was determined to express the results on a dry matter basis thus comparing them with literature references. The results obtained for the determination of the moisture content are summarised in Table 1. These results were in agreement with those found for grapefruit, namely 80–82% moisture in the peel at maturity stage.[24]

Table 1. Ratio of individual parts of yuzu fruits and dry matter content

	juice	peel	seeds
yield (%)	17.3±1.6	44.2±1.5	6.13±0.57
dry matter (%)	n.d.	16.7±0.1	52.5±0.4

n.d. not determined

3.1 *Juice Characterisation*

Yuzu juice constitutes the most commonly consumed part of fruits and therefore the results obtained are also important from a consumer perspective.

3.1.1 *Maturity Parameters: pH, Total Soluble Solids, Total Titratable Acidity and Sugar Content*

The maturity of a citrus fruit is an important criterion to assess its quality. The stage of maturity depends on many external (colour of the peel, the size of the fruit, the uniformity of the shape) and internal (aroma, texture, flavour, nutritional value) factors.[25] The maturity might be measured using diverse indices *i.e.* the quantity of juice, used principally for lemon or lime, total soluble solids (TSS) content and the TSS/total titratable acidity ratio, used mainly for oranges, mandarins or grapefruits.[26]

The TSS measuring is commonly used for a quick estimation of total sugar content. However, TSS also contain organic acids (citric acid, malic acid), amino acids or pectin. The TSS content can be expressed as °Brix and is highest when the citrus has reached its maturity. The total titratable acidity was expressed as citric acid, which is the dominant organic acid present in citrus juices. The pH is another way to express the acidity of the fruit. At maturity, sugar content is high and total acidity is low. Therefore, the ratio of TSS/acidity is the most important factor to assess the maturity of a citrus.[26–28] The diverse maturity indices measured for yuzu were compiled with those found in the literature for other citrus fruits in the Table 2. Yuzu juice was characterised by the pH of 2.8, the total titratable acidity of 3.86% citric acid and the total soluble solids of 8.36 °Brix. Yuzu juice had pH higher than lemon juice but lower than all other citrus juices.[20,21,29] On the other hand, according to the literature, the total titratable acidity of lemon and lime was higher than that of yuzu juice whereas the ratio of TSS/acidity was lower.

The major sugars present in citrus fruits are glucose, fructose and sucrose, besides minor carbohydrates such as maltose or arabinose.[28] In the yuzu juice sucrose was the predominant sugar, followed by fructose and glucose with the content of 11.2, 9.52 and 7.89 g/L, respectively. Contents are comparable with the results obtained for yuzus cultivated in different districts in Japan.[30] The glucose content was found to vary between 1.91 and 9.33 g/L with a mean value of 3.92 g/L, the fructose content ranged from 1.38 to 12.51 g/L with an average of 5.51 g/L and finally, the sucrose content was found to vary between 2.86 and 19.19 g/L with an average of 9.22 g/L. In the literature, it was reported that the ratio of sucrose, fructose and glucose is 2:1:1 for mandarin whereas lemons contain only a very small quantity of sucrose.[28,31]

Table 2. Characterisation of yuzu juice in comparison to other citrus juices based on the literature data

	pH	TSS (°Brix)	total titratable acidity (% citric acid w/w)	TSS/ acidity
yuzu	2.79±0.04	8.38±0.39	3.86±1.49	2.17
grapefruit[a]	3.00	10.3	2.07	4.09
lemon[a]	2.43	8.02	5.89	1.37
lime[b]	2.81	8.42	6.05	1.39
mandarine[cd]	3.18	10.42–16.1	0.94–1.87	8.00–14.8
orange[c]	3.41	11.8	1.69	6.97
pomelo[d]	–	10.3–11.9	0.70–0.72	14.4–17.1
sweet orange[a]	3.34	12.1	1.34	9.03
tangerine[a]	3.46	11.2	1.04	10.77

[a] ref. [29]; [b] ref. [53]; [c] ref. [20]; [d] ref. [21]

3.1.2 *Vitamin C, Organic Acids*

Vitamin C cannot be synthesised nor stored by the human body. The recommended daily intake of this essential nutrient varies between 0.015 and 0.120 g depending on the gender and the age of the person. Principal nutritional sources of vitamin C are fruits and vegetables. Vitamin C plays an important physiological role as it takes part in the production of collagen.[32] Yuzu juice analysed in this study contained 0.560 g of vitamin C per L (Table 3). This value is within the wide range of 0.08–0.72 g/L reported for different Japanese yuzu juices,[30] applying the conversion factor of 1.06 g/mL for juice density.[33] The juice obtained from yuzu cultivated in Switzerland contained nearly twice as much vitamin C as yuzu juices from two different regions of Japan.[19] The content of vitamin C of yuzu was higher than in mandarin, lime or pomelo juice but lower than in white grapefruit, sweet orange and lemon juice.[34]

Organic acids were identified and quantified by HPLC. Two main acids, namely citric and malic acids were found. The results obtained were consistent with those reported by Yu *et al.*,[30] who found values between 30.74 to 50.35 g/L for citric acid, and 2.23 to 10.60 g/L for malic acid in various citrus fruits. Citric and malic acids are the most widespread organic acids in citrus fruits. Other acids like tartaric, oxalic, succinic and benzoic can be found in trace amount.[29] These organic acids contribute to the flavour and stability of citrus juice. The highest content of citric acid was reported for lemons, limes and yuzus. Yuzu fruit contained a large amount of malic acid, similar to lime and tangerine. Most of the other citrus fruits contained more than two times less malic acid.

Table 3. Content of vitamin C and organic acid in yuzu juice expressed in g/L (mean ± standard deviation) in comparison to other citrus juice based on the literature data

	vitamin C	malic acid	citric acid
yuzu	0.560±0.042	6.18±1.01	44.67±3.2
clementine[a]	0.340	1.37	11.92
grapefruit[b]	–	4.03	19.6
lemon[ab]	0.229–0.718	1.47–6.00	55.11–73.94
lime[a]	0.354	5.18	61.50
mandarin[a]	0.515	1.78	12.74
minneola[a]	0.215	1.56	19.86
pink grapefruit[a]	0.463	1.82	21.91
pomelo[a]	0.419	0.87	13.00
sour orange[b]	–	2.21	19.6
sweet orange[ab]	0.636	1.52–7.79	13.28–13.92
sweetie (oroblanco) [a]	0.622	1.07	6.89
tangerine[b]	–	5.29	9.22
white grapefruit[a]	0.580	0.09	23.05

[a] ref. [34]; [b] ref. [29]

3.2 *Polyphenols Content and Antioxidant Activity of Yuzu Juice and Peels*

3.2.1 *Identification and Quantification of Polyphenols*

Citrus fruits are known to contain polyphenols, especially flavanones in high quantities.[8,35,36] Two flavanone aglycones, namely hesperetin and naringenin, and their glycoside derivatives, hesperidin and naringin were identified and quantified (Table 4). Hesperidin was the most abundant flavanone in yuzu juice with the concentration of 64 µg/mL, followed by naringin 47 µg/mL, naringenin 3.9 µg/mL and finally hesperetin 2.6 µg/mL. Large differences in the content of flavanones in yuzu juice from different regions of Japan were reported,[19] and in general the values noted were lower than those in the present study. Large variability in the content of phenolics for different varieties of yuzu cultivated in Korea was also stated.[10] At the same time, the flavanones contents obtained for yuzu juice in the present study were within the ranges reported in the literature for various citrus juices.[37] The differences, in individual flavonoids concentrations or polyphenols profiles, are due to numerous parameters such as the origin and the variety of citrus,[5] its development stage,[38] or maturity, but also the way the polyphenols were extracted and analysed.[21] The content within a wide range from 9.3 to 399 µg/mL and from 0.8 to 230 µg/mL for hesperidin and naringin, respectively was reported for sweet orange, clementine, lemon and grapefruit juices among others. The flavanone aglycones, hesperetin and naringenin were only found in grapefruit juice in the concentrations of 7.4 and 27 µg/mL, respectively. In yuzu juice, the content of flavanone glycosides was much higher than the content of aglycone which finds confirmation in the literature regarding citrus juices.[37,39] The sugar moiety of flavanones is important for their taste. Naringin is composed of naringenin with a neohesperidose (2-*O*-α-L-rhamnosyl-D-glucoside) bound at the 7-carbon position. This sugar moiety in the flavanone molecule confers some citrus fruits, especially grapefruits, a bitter taste.[35,37] The flavanone is tasteless if the carbohydrate moiety is bound to the aglycone as a rutinose (6-*O*-α-L-rhamnosyl-D-glucoside), for example narirutin.[28] Hesperidin is more abundant in orange, lime, lemon, and tangerine where it induces juice cloudiness because of its weak solubility in water.[40]

The same four flavonoids, which were identified and quantified in the juice, were also found in the peel (Table 4). Here also, hesperidin was the most abundant followed by naringin, naringenin and hesperetin. The flavanones aglycones were found in small amounts comparing to the glycosides. They are known to be less abundant in nature.[35,41] Somewhat higher values were noted for yuzu cultivated in Korea[9,42] whereas even 10 times higher were reported by Yoo *et al.*[10] Our results obtained for the flavanone glycosides were in agreement with those recorded for different citrus varieties (lemon, kumquat, oranges, mandarin), namely concentration levels from 0.21 to 29.8 mg/g DM for naringin and from 0.10 to 29.5 mg/g DM for hesperidin.[43] The presence of naringenin was reported in only one type of citrus peel, in mandarin. The authors reported concentrations between 0.05 and 0.49 mg/g DM in different mandarin genotypes.[44] The presence of some other polyphenols was also noted in some citrus peels, namely narirutin, neohesperidin, nobiletin, tangeretin, rutin, quercetin, kampferol, *p*-coumaric and chlorogenic acids.[7,44,45]

Table 4. Flavanone content in yuzu juice and peel expressed as µg/mL and µg/g DM, respectively. Mean ± standard deviation

	juice	peel
naringin	47±12	640±38
hesperidin	64±11	1830±250
naringenin	3.9±0.6	120±10
hesperetin	2.6±0.7	60±12

DM – dry matter

3.2.2 *Antioxidant Activity Measurements*

Although the Folin-Ciocalteu reagent reducing capacity assay (FCR) is widely used for the measurement of total polyphenol content (TPC), great attention must be paid to the fact that the reagent can interact with other compounds than polyphenols, such as vitamin C, proteins, or thiols. Since these molecules are ubiquitous in most plants, it is advisable to consider the FCR assay as total reducing capacity evaluation instead of the TPC content.[46,47] The results of this assay and three other antioxidant activity assays obtained for yuzu juice and peel are shown in the Table 5. The TPC content of yuzu juice was 0.310 mg of gallic acid equivalent (GAE) per mL, whereas that of yuzu peel amounted to 4.89 mg GAE/g DM. To our knowledge no data on yuzu juice TPC have been published so far. The values of TPC reported in the literature for citrus juices vary considerably. Substantially higher TPC concentrations ranging from 0.752 mg GAE/mL to 1.56 mg GAE/mL were noted in orange, mandarin, lemon, grapefruit and pomelo juices.[21] On the other hand, the TPC content of the yuzu peel conformed to those reported by Assefa *et al.* for yuzu cultivated in Korea.[9]

Table 5. Antioxidant activity of yuzu juice and peel (mean ± standard deviation)

	juice (per mL)	peel (per g DM)
TPC (mg GAE)	0.310±0.012	4.89±0.35
DPPH (µmol TE)	2.82±0.18	14.4±2.7
ABTS (µmol TE)	3.16±0.11	46.0±2.6
ORAC (µmol TE)	62.9±9.9	927±79

DM – dry matter; TPC – total phenolic content; GAE – gallic acid equivalent; TE – Trolox equivalent

The results of Trolox Equivalent Antioxidant Capacity by 2,2-diphenyl-1-picrylhydrazyl (DPPH), Trolox Equivalent Antioxidant Capacity by 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) and Oxygen Radical Absorbance Capacity (ORAC) assays for yuzu juice were 2.82, 3.16 and 62.95 µmol TE/mL, respectively. The results obtained by DPPH and ORAC were higher than the data found in the literature for different citrus species (lemon, lime, orange, grapefruit). DPPH and ORAC ranged from 1.49 to 2.21 µmol TE/mL[48] and from 7.69 to 13.12 µmol TE/mL,[49] respectively. Concerning the antioxidant capacity of yuzu peel the value of 14.4 µmol TE/g DM was obtained with the DPPH assay and 46.0 µmol TE/g for ABTS assay. It was reported that the peels of different citrus varieties (lime, orange, mandarin) had TE measured by DPPH assay, between 26.76 and 41.27 µmol/g DM,[50] whereas by ABTS assay TE varied from 25.3 to 103 µmol/g DM (grapefruit, lemon, orange, mandarin).[51]

The environmental conditions (soil moisture, temperature, sunshine time, culture location), the post-harvest treatment (storage temperature, time, humidity), and the processing factors (duration and temperature of extraction) influence the results of antioxidant activity.[52]

4. Conclusions

Gastronomically highly valued yuzu is being cultivated in Europe. Until now, there has been no study comparing European yuzu composition with other citrus fruits. Like for most citrus fruits, the sugar ratio for yuzu juice was 2:1:1 for sucrose, fructose and glucose. Juice from yuzu showed a lower pH than juices of grapefruit, mandarin, orange, pomelo, sweet orange and tangerine. Juice of lemon had a lower, and lime a similar pH value to yuzu juice. Both showed also higher contents of citric acid and therefore higher total titratable acidity than yuzu juice. The latter, however contained large amounts of malic acid, similar to that of lime, lemon and tangerine. Most of the other citrus fruits contained more than two times less malic acid. The juice obtained from yuzu cultivated in Switzerland contained nearly twice as much vitamin C as yuzu juices from different regions of Japan. The content of vitamin C of yuzu was higher than in mandarin, lime or pomelo juice. Hesperidin was the most abundant flavanone in yuzu juice, followed by naringin, naringenin and hesperetin. Large differences in the content of flavanones in yuzu juice from different regions of Japan were reported, in general the values noted were lower than those in the present study.

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