Full Length Research Paper

# Comparison of volatile components of flower, leaf, peel and juice of 'Page' mandarin [(Citrus *reticulata* var 'Dancy' × Citrus paradisi var 'Duncan') × Citrus clementina]

# Behzad Babazadeh Darjazi

Department of Plant Production, Faculty of Agriculture, Roudehen Branch, Islamic Azad University (I.A.U), Roudehen, Iran. E-mail: babazadeh.b@gmail.com. Tel: +98 21 33009743.

Accepted 7 July, 2011

The volatile flavor components of flower, leaf, peel and juice of 'Page' mandarin were investigated in this study. Flower components were extracted by using ultrasound (US) water bath apparatus and then eluted by n-pentane : diethylether (1:2) solvent. Leaf flavor components were extracted by using water distillation method and then eluted by using n-hexane solvent. Juice flavor components were extracted by using poly dimethyl siloxane membranes (PDMS) and then eluted by pentane : dichloromethane (2:1). Peel flavor components were extracted by using cold-press and then eluted by using n-hexane. Then, they were all analyzed by GC-FID and GC-MS. 37 flower components, 53 leaf components, 54 peel components and 47 juice components including: aldehydes, alcohols, esters, ketones, monoterpenes, sesquiterpenes and other components were identified and quantified. The major flavor components were linalool, limonene, sabinene,  $\alpha$ -pinene,  $\beta$ -myrcene,  $\delta$ -3-carene, terpinene-4-ol and  $\alpha$ -terpineol. The flower oil showed the highest content of aldehydes and alcohols. Since the aldehyde content of citrus oil is considered as one of the most important indicators of high quality, organ apparently has a profound influence on P ge mandarin oil quality.

**Key words:** 'Page' mandarin flower, 'Page' mandarin leaf, 'Page' mandarin peel, 'Page' mandarin juice, flavor components, ultrasound, water-distillation, pervaporation, cold-press.

# INTRODUCTION

Citrus hybrids have a lot of variables as a result of hybridization of many fine-quality mandarins and grapefruits, and many of these varieties are now being used successfully for juice production and as fresh fruit (Fotouhi Ghazvini and Fattahi moghadam, 2007). 'Page' mandarin that resulted from a cross between 'Dancy' 'Duncan' grapefruit, and 'Clementine' tangerine, mandarin, is a hybrid citrus crop and has been regarded as a citrus fruit with potential commercial value because of its attractive and pleasant aroma (Fotouhi Ghazvini and Fattahi moghadam, 2007). In Citrus L. species, essential oils are present in special oil glands in flowers, leaves, peel and juice. These valuable essential oils are composed of many compounds including: terpenes, sesquiterpenes, aldehydes, alcohols, esters and sterols. They may also be described as mixtures of hydrocarbons, oxygenated compounds and nonvolatile residues. Essential oils of citrus are used commercially for flavoring foods, beverages, perfumes, cosmetics, medicines, etc (Salem, 2003). Up to now, numerous investigations have been performed aimed at identifying the aroma volatiles in the mandarin flower (Babazadeh Darjazi, 2011; Salem, 2003; Kharebava and Tsertsvadze, 1986; Yoshikawa et al., 1996), leaf (Salem, 2003; Lota et al., 2000, 2001; Ekundayo et al., 1990), peel (Lota et al., 2000, 2001; Babazadeh Darjazi, 2009; Babazadeh Darjazi et al., 2009) and juice (Babazadeh Darjazi, 2009; Babazadeh Darjazi et al., 2009; Yajima et al., 1979). The quality of an essential oil may be calculated from the quantity of oxygenated compounds present in the oil

(Babazadeh Darjazi et al., 2009). Branched aldehydes and alcohols are important flavor compounds in many food products

(Salem, 2003).

Various studies have shown that the tangerine-like smell was also suggested to be mainly based on carbonyl compounds, such as  $\alpha$ -sinensal, geranial, citronellal, decanal and perilaldehyde (Salem, 2003; Babazadeh Darjazi, 2009). The quality of a honey may be calculated from the amount of oxygenated components present in the honey (Alissandrakis et al., 2003; Alistair et al., 1993) and various flowers may influence the quality of volatile flavor components present in the honey. It had been recognized previously that oxygenated compounds are important factor in deceiving and attracting the pollinators. These results may have consequences in yield in agricultural (Kite et al., 1991; Andrews et al., 2007). There have been very few studies on the essential oils of page mandarin, even though citrus oil compositions have been investigated in many areas throughout the world. In this paper, we compared the volatile compounds isolated from fresh flowers, leaves, peel and juice of 'Page' mandarin with the aim of determining whether the quantity of oxygenated compounds was influenced by the organs.

## MATERIALS AND METHODS

In 1989, 'Page' mandarin trees, grafted on 'Yuzu' rootstock, were planted at  $8 \times 4 \text{ m}^2$  with three replication at Ramsar Research Station [latitude 36° 54' N, longitude 50° 40' E; Caspian Sea climate, average rainfall 970 mm per year and average temperature 16.25°C; soil was classified as loam-clay, pH range (6.9 to 7)].

## Preparation of flower and leaf sample

In the early week of June 2007, about 500 g of leaves and at least 50 g flower were collected from many parts of the same trees, located in Ramsar Research Station, early in the morning (6 to 8 am) and only during dry weather.

## Preparation of peel sample

In the last week of November 2007, at least 10 mature fruit were collected from many parts of the same trees located in Ramsar Research Station. About 150 g of Page mandarin peel of fresh mature fruit was cold-pressed and then, the oil was separated from the crude extract by centrifugation (at 4000 rpm for 15 min at 4 °C). The supernatant was dehydrated with anhydrous sodium sulfate at 5 °C for 24 h and then filtered. The oil was stored at -25 °C until analyzed. The percent of cold-pressed peel oil is shown in Table 2.

## Preparation of juice sample

In the last week of December 2007, at least 10 mature fruit were collected from many parts of the same trees located at Ramsar research station. 'Page' mandarin juice was obtained by using the Indelicator Super Automatic, Type A2 104 extractor (China). After

extraction, juice was screened to remove peel, membrane, pulp and seed pieces according to the standard operating procedure. Each juice replicate was made with 10 'Page' mandarins. Three replicates were used for the quantitative analysis (n = 3).

### Flower extraction technique

The methodology used in this study, was described by Allissandrakis et al. (2003). In order to obtain the volatile compounds from the flowers, 50 g of fresh flowers were placed in a 2000 ml spherical flask, along with 300 ml of n-pentane : diethylether (1:2). The flask was covered and then placed in an ultrasound (US) water bath apparatus for 20 min. Ultrasonic extractions were performed with an ultrasound cleaning bath-Fisatom Scientific-FS14H (Frequency of 40 KHz, nominal power 90 W and  $24 \times 14 \times 10$  cm internal dimensions water bath). The temperature of the US water bath was held constant at  $25^{\circ}$ C. The extract was subsequently filtered through MgSO<sub>4</sub> monohydrate. The extract was finally concentrated with a gentle stream of nitrogen to 1 ml, placed in a vial and sealed. It was kept in the freezer at  $-4^{\circ}$ C until the GC-MS analysis.

#### Leaf extraction technique

In order to obtain the volatile compounds from the leaf, 500 g of fresh leaves were subjected to hydro distillation for 3 h using a Clavenger-type apparatus. N-hexane was used to isolate the oil layer from the aqueous phase. The hexane layer was dried over anhydrous sodium sulphate and stored at  $-4 \,^{\circ}$ C until used.

## Juice extraction technique

The samples were passed through a PDMS membrane (poly dimethyl siloxane) in the plate and frame geometry with a total area of 0.257 m<sup>2</sup>, namely pervap 1060 which contains corporate silicates for isolation of flavor compounds (Table 3). Extraction was done in triplicate. The flavor compounds were eluted successively with 50 ml of pentane; dichloromethane (2:1, v/v). The elute was dried over anhydrous sodium sulfate. A volume of 1 µl of the pervaporated sample was injected into GC-MS for analysis.

## GC and GC-MS

An Agilent 6890 N gas chromatograph (USA) equipped with a DB-5  $(30 \text{ m} \times 0.25 \text{ mm i.d}; \text{ film thickness} = 0.25 \mu\text{m})$  fused silica capillary column (J&W Scientific) and a flame ionization detector (FID) was used. The column temperature was programmed from 50 °C (2 min) to 188°C (20 min) at a rate of 3°C/min. The injector and detector temperatures were 220 ℃ and helium was used as the carrier gas at a flow rate of 0.8 ml/min and a linear velocity of 22 cm/s. The linear retention indices (LRIs) were calculated for all volatile components using a homologous series of n-alkanes (C9-C22) under the same GC conditions. The weight percent of each peak was calculated according to the response factor to the FID. Some standards such as citronellal, decanal,  $\beta$ -sinensal,  $\alpha$ -sinensal, linalool, terpinene-4-ol, α-terpineol, (E) nerolidol, linalyl acetate, geranyl acetate, cis-jasmone, sabinene and limonene were acquired from Sigma-Aldrich. Gas chromatography-mass spectrometry was used to identify the volatile components. The analysis was carried out with a Varian Saturn 2000R. 3800 GC linked with a Varian Saturn 2000R MS.

The oven condition, injector and detector temperatures, and column (DB-5) were the same as those given earlier for the Agilent 6890 N GC. Helium was the carrier gas at a flow rate of 1.1 ml/min

and a linear velocity of 38.7 cm/s. Injection volume was 1  $\,\mu\text{L}.$ 

#### Identification of components

Components were identified by comparing their LRIs and matching their mass spectra with those of reference compounds in the data system of the Wiley library and NIST Mass Spectral Search program (Chem. SW. Inc; NIST 98 version database) connected to a Varian Saturn 2000R MS. Identifications were also determined by comparing the retention time of each compound with that of known compounds (Adams, 2001; McLafferty and Stauffer, 1991).

# RESULTS

## Flavor compounds of the 'Page' mandarin flower

GC-MS analysis of the flavor compounds extracted from Page mandarin flower by using ultrasound water bath allowed the identification of 37 volatile components (Table 1 and Figure 1): 17 oxygenated terpenes (3 aldehydes, 13 alcohols and one ketone), 14 non oxygenated terpenes (9 monoterpens and 5 sesgiterpens) and six other components.

## Flavor compounds of the 'Page' mandarin leaf

GC-MS analysis of the flavor compounds extracted from 'Page' mandarin leaf by using water distillation allowed identification of 53 volatile components (Table 1): 26 oxygenated terpenes (8 aldehydes, 15 alcohols and 3 esters], 26 non oxygenated terpenes (15 monoterpens and 11 sesqiterpens) and one other component.

## Flavor compounds of the 'Page' mandarin peel

GC-MS analysis of the flavor compounds extracted from 'Page' mandarin peel by using cold-press allowed identification of 54 volatile components (Table 1): 25 oxygenated terpenes [11 aldehydes, 9 alcohols, 3 esters and 2 ketones], 29 non oxygenated terpenes [12 monoterpens and 17 sesqiterpens].

## Flavor compounds of the 'Page' mandarin juice

GC-MS analysis of the flavor compounds extracted from 'Page' mandarin juice by using PDMS membranes allowed identification of 47 volatile components (Table 1): 22 oxygenated terpenes [12 aldehydes, 8 alcohols, one esters and one ketones], 25 non oxygenated terpenes [12 monoterpens and 13 sesqiterpens].

## Aldehydes

16 aldehyde components that were identified in this analysis were: octanal, citronellal, decanal, 3- cyclohexene

-1-acetaldehyde, neral, (E)-2-decanal, geranial, perillaldehyde, undecanal, (E)2,4-decadienal, 5-dodecen-1-al, 7-dodecen-1-al, dodecanal, tetradecanal, β-sinensal and  $\alpha$ -sinensal (Table 2). In addition, they were quantified from 0.44 to 7.16%, that it was determined and reported as relative amount of these compounds in oil in this study. These findings were similar to previous studies (Salem, 2003; Babazadeh Dariazi. 2009. 2011; Babazadeh Darjazi et al., 2009). Tangerine oil is easily distinguished from other citrus oils by its content of aliphatic aldehydes. Two main various aliphatic aldehydes were  $\beta$ -sinensal and  $\alpha$ -sinensal. In addition, tangerine oil also contained citronellal (Salem, 2003). βsinensal has a woody aroma (Sawamura et al., 2004), and is considered as one of the major contributors to mandarin flavor (Salem, 2003).

Since the aldehyde content of citrus oil is considered as one of the most important indicators of high quality, organ apparently has a profound influence on 'Page' mandarin oil quality. Among the four organs examined, flower showed the highest content of aldehydes (Table 2).

Flower aldehydes were also compared with those of leaf, peel and juice in this study.  $\beta$ -sinensal and  $\alpha$ -sinensal were identified in flower and leaf oil, while they were not detected in peel and juice. When compared with juice, the flower improved and increased aldehyde components about 16 times for Page mandarin (Table 2).

# Alcohols

25 alcohol components identified in this study were linalool, phenyl ethyl alcohol, P-mentha-trans-2,8-dien-1ol, terpinene-1- ol, isopulegol, terpinene-4-ol,  $\alpha$ -terpineol, myrtenol, (z)-piperitol, lilace alcohol B,  $\beta$ -citronellol, lilace alcohol D, cis-carveol, geraniol, Perillalcohol, indol, Pmenth-1-en-9-ol, 2,6-dimethyl-2,7-octadien-1,6-diol, elemol, (E) nerolidol, germacrene D-4-ol, spathulenol,  $\alpha$ muurolol,  $\alpha$ -cadinol and E,E-cis-farnesol (Table 2).

The total amount of alcohols ranged from from 0.99 to 39.27% and it was determined and reported as relative amount of those compounds in 'Page' mandarin oil. Linalool was the primary component in this study and it was the most abundant. Linalool, the most significant alcohol compound of mandarin oil, is recognized as being very important to good mandarin flavor (Salem, 2003; Babazadeh Darjazi, 2009). Linalool has a flowery (rose-like) aroma (Sawamura et al., 2004) and its level is important to flavor character in mandarin flower, leaf, peel and juice (Salem, 2003; Babazadeh Darjazi, 2009). Among the four organs examined, flower showed the highest content of alcohols (Table 2).

Flower alcohols were also compared to those of leaf, peel and juice in this study. Lilace alcohol B, lilace alcohol D, indol, 2,6-dimethyl-2,7-octadien-1,6-diol, E,Ecis-farnesol and phenyl ethyl alcohol were identified in flower oil, while they were not detected in leaf, peel and juice. When compared with juice, the flower Table 1. Chemical composition of essential oils of the flower, leaf, peel and juice of Page mandarin.

| S/N | S/N Component                |   | Leaf | Peel | Juice | KI   | KI S/N Component |                                     | Flower | Leaf | Peel | Juice | KI   |
|-----|------------------------------|---|------|------|-------|------|------------------|-------------------------------------|--------|------|------|-------|------|
| 1   | α- thujene                   |   | *    |      | *     | 930  | 52               | Citronellyl acetate                 |        | *    |      |       | 1353 |
| 2   | α - Pinene                   | * | *    | *    | *     | 939  | 53               | Unknown                             |        |      | *    | *     | 1353 |
| 3   | Camphene                     |   | *    | *    | *     | 954  | 54               | Neryl acetate                       |        | *    |      |       | 1362 |
| 4   | Sabinene                     | * | *    | *    | *     | 975  | 55               | 2,6-dimethyl-2,7-octadiene-1,6-diol | *      |      |      |       | 1366 |
| 5   | β - Pinene                   | * |      | *    |       | 979  | 56               | α-copaene                           |        |      | *    | *     | 1377 |
| 6   | β -myrcene                   | * | *    | *    | *     | 991  | 57               | β-patchulene                        |        |      | *    |       | 1381 |
| 7   | Octanal                      |   |      | *    | *     | 999  | 58               | Geranyl acetate                     |        | *    |      |       | 1381 |
| 8   | α - phellandrene             |   | *    | *    | *     | 1003 | 59               | 5-dodecen-1-al                      |        |      |      | *     | 1390 |
| 9   | δ-3–carene                   | * | *    | *    | *     | 1031 | 60               | β-elemene                           |        |      | *    | *     | 1391 |
| 10  | α - terpinene                |   | *    |      |       | 1017 | 61               | Cis-jasmone                         | *      |      |      |       | 1391 |
| 11  | Limonene                     | * | *    | *    | *     | 1029 | 62               | 7-dodecen-1-al                      |        |      | *    |       | 1395 |
| 12  | (Z)- β - ocimene             |   | *    | *    | *     | 1037 | 63               | Dodecanal                           |        | *    | *    | *     | 1409 |
| 13  | (E)- β - ocimene             | * | *    |      |       | 1055 | 64               | (Z)- β-caryophyllene                | *      | *    | *    | *     | 1409 |
| 14  | γ- terpinene                 |   | *    |      |       | 1060 |                  | Limonene-10-yl acetate              |        |      | *    |       | 1414 |
| 15  | (E)-sabinene hydrate         | * | *    |      |       | 1070 | 66               | P-Menth-1-en-9-ol acetate           |        |      | *    |       | 1423 |
| 16  | Unknown                      |   |      | *    | *     | 1075 | 67               |                                     |        | *    | *    | *     | 1437 |
| 17  | α -terpinolene               | * | *    | *    | *     | 1089 | 68               | α-guaiene                           |        |      | *    |       | 1440 |
| 18  | Linalool                     | * | *    | *    | *     | 1097 | 69               | Aromadendrene                       |        |      |      | *     | 1441 |
| 19  | Phenyl ethyl alcohol         | * |      |      |       | 1107 | 70               | (Z)- β-farnesene                    | *      | *    | *    |       | 1443 |
| 20  | P-mentha-trans-2,8-dien-1-oi |   |      |      | *     | 1128 | 71               | α - humulene                        |        | *    | *    | *     | 1455 |
| 21  | Allo ocimene                 |   | *    |      |       | 1132 | 72               | α - amorphene                       |        | *    | *    |       | 1485 |
| 22  | Terpinene-1-ol               |   | *    |      |       | 1134 | 73               | Germacrene D                        |        | *    | *    | *     | 1485 |
| 23  | Cis-limonene oxide           |   |      | *    | *     | 1137 | 74               | Bicyclogermacrene                   |        | *    | *    |       | 1500 |
| 24  | Trans-limonene oxide         |   |      | *    | *     | 1142 | 75               | α-muurolene                         |        |      | *    | *     | 1500 |
| 25  | Citronellal                  |   | *    | *    | *     | 1153 | 76               | δ-guaiene                           |        |      | *    | *     | 1503 |
| 26  | Isopulegol                   |   | *    |      |       | 1160 | 77               | E,E, α-farnesene                    | *      | *    |      | *     | 1506 |
| 27  | Terpinen-4-ol                | * | *    | *    | *     | 1177 | 78               | y-cadinene                          |        |      | *    |       | 1514 |
| 28  | α - terpineol                | * | *    | *    | *     | 1189 | 79               | β-sesquiphellandrene                | *      | *    |      |       | 1523 |
| 29  | Myrtenol                     |   | *    |      |       | 1196 | 80               | δ-cadinene                          |        |      | *    | *     | 1523 |
| 30  | Decanal                      | * | *    | *    | *     | 1207 | 81               | Elemol                              |        | *    | *    | *     | 1550 |
| 31  | (z)-piperitol                |   | *    |      |       | 1208 | 82               | (E)–nerolidol                       | *      | *    | *    |       | 1563 |
| 32  | Lilac alcohol B              | * |      |      |       | 1217 | 83               | Germacrene B                        |        |      | *    | *     | 1571 |
| 33  | 3-cyclohexene-1-acetaldehyde |   |      | *    | *     | 1224 | 84               | Germacrene D– 4–ol                  | *      |      | *    | *     | 1576 |
| 34  | β - citronellol              |   | *    | *    |       | 1226 | 85               | Spathulenol                         | *      | *    |      |       | 1578 |
| 35  | Cis-carveol                  |   | *    |      |       | 1229 | 86               | Caryophyllene oxide                 |        | *    |      |       | 1583 |
| 36  | Lilac alcohol D              | * |      |      |       | 1233 |                  | Unknown                             | *      |      |      |       | 1586 |

#### Table 1 Contd.

| 37 | Neral                                   |   | * | * | * | 1238 | 88  | Hexadecane       | * |   |   |   | 1600 |
|----|---|---|---|---|---|------|-----|------------------|---|---|---|---|------|
| 38 | Carvone                                 |   |   | * | * | 1243 | 89  | Tetradecanal     |   |   |   | * | 1613 |
| 39 | Geraniol                                |   | * |   |   | 1253 | 90  | Unknown          |   |   | * | * | 1634 |
| 40 | Linalyl acetate                         |   |   | * | * | 1257 | 91  | α -muurolol      |   | * |   |   | 1646 |
| 41 | (E)-2-decanal                           |   |   |   | * | 1263 | 92  | α –cadinol       | * | * |   |   | 1654 |
| 42 | Geranial                                |   | * | * |   | 1267 | 93  | 8-heptadecene    | * |   |   |   | 1676 |
| 43 | Perillaldehyde                          |   |   | * | * | 1272 | 94  | heptadecane      | * |   |   |   | 1700 |
| 44 | 3,3,5-trimethyl cyclohex-2-en-<br>1-one |   |   | * |   | 1278 | 95  | β-sinensal       | * | * |   |   | 1700 |
| 45 | Indol                                   | * |   |   |   | 1291 | 96  | E,E-cis-farnesol | * |   |   |   | 1725 |
| 46 | Perillalcohol                           |   |   | * | * | 1295 | 97  | α-sinensal       | * | * |   |   | 1757 |
| 47 | P-menth-1-en-9-ol                       |   |   | * | * | 1295 | 98  | Unknown          | * |   |   |   | 1832 |
| 48 | Undecanal                               |   |   | * | * | 1307 | 99  | Caffeine         |   | * |   |   | 1842 |
| 49 | (E)-2,4-decadienal                      |   | * | * | * | 1317 | 100 | Nonadecane       | * |   |   |   | 1900 |
| 50 | δ- elemene                              | * | * | * | * | 1338 | 101 | Eicosane         | * |   |   |   | 2000 |
| 51 | Unknown                                 | * |   |   |   | 1341 | 102 | Heneicosane      | * |   |   |   | 2100 |

\*There is in oil

improved and increased alcohol components about 39 times for Page mandarin (Table 2).

#### Esters

Six ester components identified in the analysis were linalyl acetate, citronellyl acetate, neryl acetate, geranyl acetate, limonene-10-yl acetate and P-menth-1-en-9-yl acetate (Salem, 2003; Babazadeh Darjazi et al., 2009). The total amount of esters ranged from 0.00 to 1.63% in oil and the geranyl acetate was the most abundant. Among the four organs examined, leaf showed the highest content of esters in oil (Table 2).

#### Ketone

Three ketone compound identified in the analysis were carvone, 3,5,5-trimethyl cyclohex-2-en-1-one

and cis-jasmone. Among the four organs examined, flower showed the highest content of ketones (Table 2).

Flower ketones were also compared with those of leaf, peel and juice in this study. Cis-jasmone was identified in flower oil, and it was not detected in leaf, peel and juice (Table 2).

## Monoterpenes hydrocarbons

The total amount of monoterpene hydrocarbons ranged from 31.58 to 96.88% in the oil. Sabinene was the major component among the monoterpene hydrocarbons of 'Page' mandarin flower and leaf oil, while limonene was the major component among the monoterpene hydrocarbons of 'Page' mandarin peel oil and juice. Limonene has a weak citrus-like aroma (Sawamura et al., 2004) and is considered as one of the major contributors to mandarin flavor (Salem, 2003).

Among the four organs examined, juice had the highest monoterpenes hydrocarbons in oil (Table 2).

## Sesquiterpenes hydrocarbons

The total amount of sesquiterpene hydrocarbons ranged from 0.66 to 9.28% in the oil. (Z)- $\beta$  -farnesene and E, E- $\alpha$ -farnesene were the major components among the sesquiterpen hydrocarbons of 'Page' mandarin flower oil. Among the four organs examined, flower had the highest sesquiterpenes content (Table 2).

## DISCUSSION

Our observations that changing organ has an effect on some of the components of

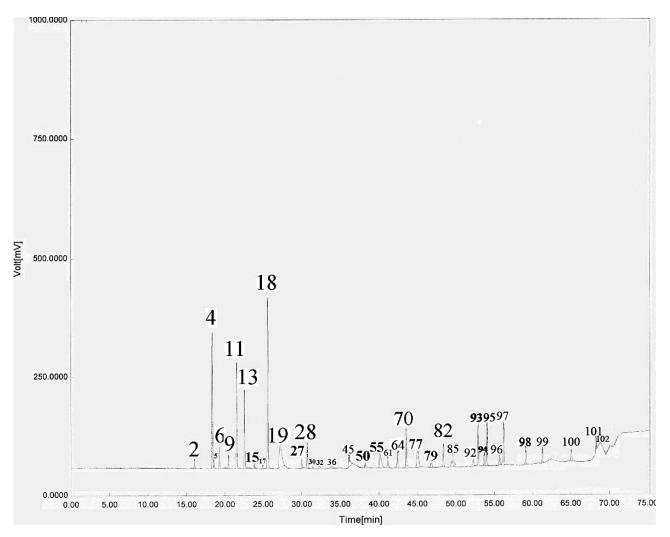




Table 2. Comparison of volatile components of flower, leaf, peel and juice of Page mandarin.

| Compound                        | Flo  | wer    | Le   | eaf    | Pe   | el     | Juice |        |
|---------------------------------|------|--------|------|--------|------|--------|-------|--------|
| Compound                        | Mean | St.err | Mean | St.err | Mean | St.err | Mean  | St.err |
| Oxygenated compound             |      |        |      |        |      |        |       |        |
| a) Aldehyds                     |      |        |      |        |      |        |       |        |
| 1) Octanal                      | 0.00 | 0.00   | 0.00 | 0.00   | 0.12 | 0.00   | 0.02  | 0.005  |
| 2) Citronellal                  | 0.00 | 0.00   | 2.96 | 0.03   | 0.07 | 0.01   | 0.01  | 0.005  |
| 3) Decanal                      | 0.16 | 0.04   | 0.09 | 0.009  | 0.58 | 0.01   | 0.26  | 0.0005 |
| 4) 3-cyclohexene-1-acetaldehyde | 0.00 | 0.00   | 0.00 | 0.00   | 0.11 | 0.005  | 0.02  | 0.005  |
| 5) Neral                        | 0.00 | 0.00   | 0.01 | 0.005  | 0.03 | 0.01   | 0.009 | 0.0005 |
| 6) (E)2,4-decanal               | 0.00 | 0.00   | 0.00 | 0.00   | 0.00 | 0.00   | 0.01  | 0.005  |
| 7) Geranial                     | 0.00 | 0.00   | 0.01 | 0.001  | 0.04 | 0.01   | 0.00  | 0.00   |
| 8) Perillaldehyde               | 0.00 | 0.00   | 0.00 | 0.00   | 0.04 | 0.005  | 0.02  | 0.00   |
| 9) Undecanal                    | 0.00 | 0.00   | 0.00 | 0.00   | 0.03 | 0.00   | 0.01  | 0.005  |
| 10) (E)2,4-decadienal           | 0.00 | 0.00   | 0.15 | 0.04   | 0.09 | 0.02   | 0.03  | 0.00   |
| 11) 5-dodecen-1-al              | 0.00 | 0.00   | 0.00 | 0.00   | 0.00 | 0.00   | 0.008 | 0.001  |
| 12) 7-dodecen-1-al              | 0.00 | 0.00   | 0.00 | 0.00   | 0.02 | 0.01   | 0.00  | 0.00   |
| 13) Dodecanal                   | 0.00 | 0.00   | 0.07 | 0.001  | 0.21 | 0.02   | 0.05  | 0.005  |

# Table 2 Contd.

| 14) Tetradecanal                               | 0.00  | 0.00 | 0.00  | 0.00  | 0.00 | 0.00  | 0.0008 | 0.0002 |
|--|-------|------|-------|-------|------|-------|--------|--------|
| 15) β-sinensal                                 | 3.20  | 0.28 | 3.03  | 0.04  | 0.00 | 0.00  | 0.00   | 0.00   |
| 16) α-sinensal                                 | 3.80  | 0.30 | 0.64  | 0.02  | 0.00 | 0.00  | 0.00   | 0.00   |
| Total  | 7.16  | 0.62 | 6.96  | 0.14  | 1.34 | 0.10  | 0.44   | 0.03   |
| b) Alcohols                                    |       |      |       |       |      |       |        |        |
| 1) Linalool                                    | 16.82 | 0.61 | 21.47 | 0.08  | 1.24 | 0.08  | 0.84   | 0.01   |
| <ol><li>Phenyl ethyl alcohol</li></ol>         | 8.16  | 0.96 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| <ol><li>P-Mentha-trans-2,8-dien-1-ol</li></ol> | 0.00  | 0.00 | 0.00  | 0.00  | 0.00 | 0.00  | 0.002  | 0.00   |
| 4) Terpinene-1-ol                              | 0.00  | 0.00 | 0.08  | 0.01  | 0.00 | 0.00  | 0.00   | 0.00   |
| 5) Isopulegol                                  | 0.00  | 0.00 | 0.10  | 0.01  | 0.00 | 0.00  | 0.00   | 0.00   |
| 6) Terpinene-4-ol                              | 0.60  | 0.05 | 2.56  | 0.04  | 0.01 | 0.00  | 0.005  | 0.000  |
| 7) α- terpineol                                | 2.19  | 0.16 | 0.38  | 0.007 | 0.10 | 0.02  | 0.07   | 0.00   |
| 8) Myrtenol                                    | 0.00  | 0.00 | 0.03  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| 9) (Z)-piperitol                               | 0.00  | 0.00 | 0.09  | 0.009 | 0.00 | 0.00  | 0.00   | 0.00   |
| 10) Lilac alcohol B                            | 0.38  | 0.03 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| 11) β-citronellol                              | 0.00  | 0.00 | 0.57  | 0.01  | 0.04 | 0.01  | 0.00   | 0.00   |
| 12) Lilac alcohol D                            | 0.43  | 0.12 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| 13) Cis-carveol                                | 0.00  | 0.00 | 0.54  | 0.02  | 0.00 | 0.00  | 0.00   | 0.00   |
| 14) Geraniol                                   | 0.00  | 0.00 | 0.43  | 0.01  | 0.00 | 0.00  | 0.00   | 0.00   |
| 15) Perillalcohol                              | 0.00  | 0.00 | 0.00  | 0.00  | 0.05 | 0.01  | 0.02   | 0.005  |
| 16) Indol                                      | 1.81  | 0.16 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| 17) P-Menth-1-en-9-ol                          | 0.00  | 0.00 | 0.00  | 0.00  | 0.02 | 0.00  | 0.008  | 0.001  |
| 18) 2,6-dimethyl-2,7-octadien-1,6-diol         | 3.42  | 0.55 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| 19) Elemol                                     | 0.00  | 0.00 | 0.008 | 0.001 | 0.28 | 0.05  | 0.04   | 0.005  |
| 20) (E)-nerolidol                              | 2.00  | 0.30 | 0.29  | 0.008 | 0.09 | 0.04  | 0.00   | 0.00   |
| 21) Germacrene-D-4-ol                          | 0.49  | 0.05 | 0.00  | 0.00  | 0.03 | 0.005 | 0.009  | 0.005  |
| 22) Spathulenol                                | 1.39  | 0.27 | 0.58  | 0.01  | 0.00 | 0.00  | 0.00   | 0.00   |
| 23) α-muurolol                                 | 0.00  | 0.00 | 0.10  | 0.01  | 0.00 | 0.00  | 0.00   | 0.00   |
| 24) α-cadinol                                  | 0.79  | 0.29 | 0.12  | 0.001 | 0.00 | 0.00  | 0.00   | 0.00   |
| 25) E,E-cis-farnesol                           | 0.79  | 0.29 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| Total  | 39.27 | 3.84 | 27.34 | 0.22  | 1.86 | 0.21  | 0.99   | 0.02   |
| c) Esters                                      |       |      |       |       |      |       |        |        |
| 1) Linalyl acetate                             | 0.00  | 0.00 | 0.00  | 0.00  | 0.01 | 0.005 | 0.005  | 0.001  |
| 2) Citronellyl acetate                         | 0.00  | 0.00 | 0.09  | 0.001 | 0.00 | 0.00  | 0.00   | 0.00   |
| 3) Neryl acetate                               | 0.00  | 0.00 | 0.22  | 0.008 | 0.00 | 0.00  | 0.00   | 0.00   |
| 4) Granyl acetate                              | 0.00  | 0.00 | 1.32  | 0.02  | 0.00 | 0.00  | 0.00   | 0.00   |
| 5) Limonene-10-yl acetate                      | 0.00  | 0.00 | 0.00  | 0.00  | 0.10 | 0.01  | 0.00   | 0.00   |
| <ol><li>P-Menth-1-en-9-yl acetate</li></ol>    | 0.00  | 0.00 | 0.00  | 0.00  | 0.03 | 0.005 | 0.00   | 0.00   |
| Total  | 0.00  | 0.00 | 1.63  | 0.02  | 0.14 | 0.02  | 0.005  | 0.001  |
| d) Ketones                                     |       |      |       |       |      |       |        |        |
| 1) Carvone                                     | 0.00  | 0.00 | 0.00  | 0.00  | 0.06 | 0.01  | 0.01   | 0.00   |
| 2) 3,5,5-trimethyl cyclohex-2-en-1-one         | 0.00  | 0.00 | 0.00  | 0.00  | 0.11 | 0.01  | 0.00   | 0.00   |
| 3) Cis-jasmone                                 | 0.79  | 0.29 | 0.00  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00   |
| Total  | 0.79  | 0.29 | 0.00  | 0.00  | 0.17 | 0.02  | 0.01   | 0.00   |
| Monoterpenes                                   |       |      |       |       |      |       |        |        |
| 1) α-Thujene                                   | 0.00  | 0.00 | 0.39  | 0.01  | 0.00 | 0.00  | 0.002  | 0.00   |
| 2) α-Pinene                                    | 0.60  | 0.05 | 1.72  | 0.03  | 0.31 | 0.01  | 0.51   | 0.005  |
| 3) Camphene                                    | 0.00  | 0.00 | 0.07  | 0.001 | 0.01 | 0.00  | 0.003  | 0.003  |

# Table 2 Contd.

| 4) Sabinene                | 11.81 | 0.97  | 35.91 | 0.23  | 0.19  | 0.01  | 0.27   | 0.00  |
|----------------------------|-------|-------|-------|-------|-------|-------|--------|-------|
| 5) β-Pinene                | 0.60  | 0.05  | 0.00  | 0.00  | 0.01  | 0.00  | 0.00   | 0.00  |
| 6) β-Myrcene               | 1.58  | 0.22  | 2.48  | 0.02  | 1.93  | 0.03  | 2.09   | 0.01  |
| 7) α -Phellandrene         | 0.00  | 0.00  | 0.27  | 0.02  | 0.03  | 0.005 | 0.05   | 0.00  |
| 8) δ -3-Carene             | 0.79  | 0.31  | 3.24  | 0.01  | 0.03  | 0.01  | 0.09   | 0.01  |
| 9) α-Terpinene             | 0.00  | 0.00  | 0.71  | 0.01  | 0.00  | 0.00  | 0.00   | 0.00  |
| 10) Limonene               | 9.01  | 0.45  | 4.13  | 0.04  | 87.45 | 0.49  | 93.82  | 0.09  |
| 11) (Z)- β-Ocimene         | 0.00  | 0.00  | 0.24  | 0.01  | 0.77  | 0.04  | 0.04   | 0.02  |
| 12) (E)- β-Ocimene         | 6.38  | 0.40  | 6.27  | 0.01  | 0.00  | 0.00  | 0.00   | 0.00  |
| 13) γ-Terpinene            | 0.00  | 0.00  | 1.25  | 0.02  | 0.00  | 0.00  | 0.00   | 0.00  |
| 14) (z) Sabinene hydrate   | 0.41  | 0.03  | 0.55  | 0.01  | 0.00  | 0.00  | 0.00   | 0.00  |
| 15) α-terpinolene          | 0.40  | 0.003 | 0.82  | 0.03  | 0.02  | 0.01  | 0.0005 | 0.00  |
| 16)Allo ocimene            | 0.00  | 0.00  | 0.24  | 0.01  | 0.00  | 0.00  | 0.00   | 0.00  |
| 17) Cis-limonene oxide     | 0.00  | 0.00  | 0.00  | 0.00  | 0.01  | 0.005 | 0.006  | 0.000 |
| 18) Trans-limonene oxide   | 0.00  | 0.00  | 0.00  | 0.00  | 0.01  | 0.005 | 0.008  | 0.00  |
| Total                      | 31.58 | 2.48  | 58.29 | 0.46  | 90.77 | 0.61  | 96.88  | 0.13  |
| Sesquiterpenes             |       |       |       |       |       |       |        |       |
| 1) δ-elemene               | 0.55  | 0.02  | 0.26  | 0.003 | 0.48  | 0.05  | 0.15   | 0.01  |
| 2) β-patchulene            | 0.00  | 0.00  | 0.00  | 0.00  | 0.01  | 0.005 | 0.00   | 0.00  |
| 3) α-copaene               | 0.00  | 0.00  | 0.00  | 0.00  | 0.10  | 0.02  | 0.03   | 0.00  |
| 4) β-elemene               | 0.00  | 0.00  | 0.00  | 0.00  | 0.02  | 0.01  | 0.07   | 0.00  |
| 5) (Z)-β-Caryophyllene     | 1.20  | 0.11  | 0.96  | 0.008 | 0.03  | 0.005 | 0.01   | 0.00  |
| 6) γ-Elemene               | 0.00  | 0.00  | 0.05  | 0.001 | 0.13  | 0.01  | 0.02   | 0.00  |
| 7) α-Guaiene               | 0.00  | 0.00  | 0.00  | 0.00  | 0.01  | 0.02  | 0.00   | 0.00  |
| 8) Aromadendrene           | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.04   | 0.00  |
| 9) (Z)-β-Farnesene         | 3.39  | 0.06  | 1.16  | 0.01  | 0.17  | 0.06  | 0.00   | 0.00  |
| 10) α-Humulene             | 0.00  | 0.00  | 0.10  | 0.001 | 0.18  | 0.01  | 0.04   | 0.00  |
| 11) α-Amorphen             | 0.00  | 0.00  | 0.01  | 0.00  | 0.03  | 0.02  | 0.00   | 0.00  |
| 12) Germacrene D           | 0.00  | 0.00  | 0.05  | 0.001 | 0.82  | 0.07  | 0.20   | 0.02  |
| 13) Bicyclogermacrene      | 0.00  | 0.00  | 1.04  | 0.01  | 0.03  | 0.01  | 0.00   | 0.00  |
| 14) α-Muurolene            | 0.00  | 0.00  | 0.00  | 0.00  | 0.12  | 0.02  | 0.02   | 0.00  |
| 15) δ-Guaiene              | 0.00  | 0.00  | 0.00  | 0.00  | 0.08  | 0.01  | 0.008  | 0.000 |
| 16) E, E, α-Farnesene      | 3.58  | 0.33  | 0.009 | 0.00  | 0.00  | 0.00  | 0.004  | 0.00  |
| 17) γ-Cadinene             | 0.00  | 0.00  | 0.00  | 0.00  | 0.02  | 0.01  | 0.00   | 0.00  |
| 18) β-Sesquiphellandrene   | 0.56  | 0.04  | 0.35  | 0.008 | 0.00  | 0.00  | 0.00   | 0.00  |
| 19) δ-Cadinene             | 0.00  | 0.00  | 0.00  | 0.00  | 0.20  | 0.01  | 0.05   | 0.00  |
| 20) Germacrene B           | 0.00  | 0.00  | 0.00  | 0.00  | 0.03  | 0.01  | 0.02   | 0.00  |
| 21) Caryophyllene oxide    | 0.00  | 0.00  | 0.25  | 0.003 | 0.00  | 0.00  | 0.00   | 0.00  |
| Total                      | 9.28  | 0.56  | 4.23  | 0.04  | 2.46  | 0.35  | 0.66   | 0.05  |
| Other compounds            |       |       |       |       |       |       |        |       |
| 1) Hexadecane              | 0.38  | 0.03  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| 2) 8-heptadecene           | 4.00  | 0.03  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| 3) Heptadecane             | 0.98  | 0.27  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| 4) Caffeine                | 0.00  | 0.00  | 0.01  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| 5) Nonadecane              | 1.20  | 0.11  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| 6) Eicosane                | 0.44  | 0.03  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| 7) Heneicosane             | 1.20  | 0.11  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| Total                      | 8.20  | 0.58  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   | 0.00  |
| Total oxygenated compounds | 47.22 | 4.75  | 35.93 | 0.38  | 3.51  | 0.35  | 1.44   | 0.05  |
| Total                      | 96.28 | 8.37  | 98.46 | 0.88  | 96.74 | 1.31  | 98.98  | 0.23  |

| System          | Binary, ternary and multicomponent solutions (Me, Et, Pr, EtAc, EtBu) industrially condensed from orange juice concentration |
|-----------------|--|
| Membrane        | PDMS-1060 <sup>e</sup>   |
| Module          | PF   |
|                 | 0.002 ≤ c ≤ 13.65% <sup>b</sup>  |
| Operating range | 5≤ T ≤ 45 ℃  |
|                 | P <sub>P</sub> < 10 mmHg   |

**Table 3.** Characteristics of membrane.

<sup>e</sup>General electric Co, <sup>b</sup>Permanent-dependent value.

mandarin oil are in accordance with other observations (Salem, 2003; Babazadeh Darjazi et al., 2009; Loata et al., 2000). The compositions of Page mandarin obtained from flower, leaf, peel and juice were very similar. However, relative concentration of compounds differed according to type of materials.

When compared with juice, the flower improved and increased alcohol components about 39 times for page mandarin. The amount of alcohol components obtained from juice were low probably because of the decrease in endo-genous enzymes activity (isopentenyl pyrophosphate isomerase (IPI)and geranyl pyrophosphate synthase (GPS)] (Hay and Waterman, 1995), resulting in decreased labile compounds. Also, the lower proportion of the detected alcohol components in juice was probably due to seasonal temperature (Sekiya et al., 1984), which is the most important environmental factor in the control of endogenous enzymes.

According to our results, it appears that the relative percentages of the identified compounds depend on the plant part studied. However, it should be kept in mind that the isolation procedure was different (Gauvin et al., 2004; Loata et al., 2000) for flower (ultrasound), leaves peel (cold-press) (hydrodistillation), and juice (pervaporation). This may also influence the chemical composition of the essential oil Babazadeh Darjazi, 2011. The pronounced enhancement in the amount of oxygenated compounds, when flower was used as the organ, showed that either the synthesis of GPP is enhanced or activities of both enzymes (IPI and GPS) increased (Hay and Waterman, 1995).

## Conclusion

In this study, we found that the percentage of flavor compounds was significantly affected by organ. The essential oil obtained from the flower contained more oxygenated compounds and fewer monoterpene components than those isolated from the leaf, peel and juice. It is easy to observe the significant variations among flower and other organs, mainly in terms of the quantities of oxygenated compounds. The essential oils and their aroma compounds are very important and widely used in hygienic products, aromatherapy, pharmacy, food industries, cosmetics and other areas. Therefore, many studies, such as this, is very crucial in order to identify the type of chemical constituents that exist in the materials that are to be used, before the essential oil can be utilized in those industries. Further research on the relationship between organ and essential oil (oxygenated terpenes) is necessary.

## ACKNOWLEDGEMENT

The author would like to express his gratitude to Z. Kadkhoda from Institute of Medicinal Plants located at Supa blvd-Km 55 of Tehran–Qazvin (Iran) for her help in GC-MS and GC analysis.

#### REFERENCES

- Adams RP (2001). Identification of essential oil components by gas chromatography / mass spectrometry. Allured Publishing Corporation, Carol Stream, IL.
- Alissandrakis E, Daferera D, Tarantilis PA, Polissiou M, Harizanis PC (2003). Ultrasound assisted extraction of volatile compounds from citrus flowers and citrus honey. Food.Chem., 82: 575-582.
- Alistair LW, Yinrong LU, Seng-To Tan (1993). Extractives from New Zealand honey 4.linalool derivatives and other components from nodding thistle (Corduus nutans) honey. J. Agric. Food. Chem., 41(6): 873-878.
- Andrews ES, Theis N, Alder LS (2007). Pollinator and herbivore attraction to cucurbita floral volatiles. J. Chem. Ecol., 33: 1682-1691.
- Babazadeh Darjazi B (2009). The effects of rootstock on the volatile flavor components of page mandarin juice and peel. Ph.D of science Thesis. Islamic Azad University. Science and Research Branch.
- Babazadeh Darjazi B (2011). The effects of rootstock on the volatile flavour components of page mandarin flower and leaf. Afr. J. Agric. Res., 6(7): 1884-1896.
- Babazadeh Darjazi B (2011). A comparison of volatile components of flower of page mandarin obtained by ultrasound-assisted extraction and hydrodistillation. J. Med. Plant Res. 5(13): 2840-2847.
- Babazadeh Darjazi B, Rustaiyan A, Talaei A, Khalighi A, Larijani K, Golein B, Taghizad R (2009). The effects of rootstock on the volatile flavor components of page mandarin juice and peel. Iran.J.Chem.Chem.Eng., 28(2): 99-111.
- Ekundayo O, Bakare O, Adesomoju A, Stahl-Bisk up E (1990). Leaf volatile oil composition of mandarin (C.reticulata Blanco) from Nigeria J. Essent.Oil. Res., 2: 329-330.
- Fotouhi Ghazvini R, Fattahi moghadam J (2007). Citrus growing in Iran: pp. 40-77

- Gauvin A, Ravaomanarivo H, Smadja J (2004). Comparative analysis by gas chromatography-mass spectrometry of the essential oils from bark and leaves of Cedrelopsis grevei Baill, an aromatic and medicinal plant from Madagascar. J. Chromatogr. A., 1029: 279-282.
- Hay RKM, Waterman P (1995). Volatile oil crops; their biology, biochemistry, and production.
- Kharebava LG, Tsertsvadze VV (1986). Volatile compounds of flower of Citrus unshiu Marc. Subtrop. Kult., (1): 119-121.
- Kite G, Reynolds T, Prance T (1991). Potential pollinator –attracting chemicals from Victoria (Nymphaeaceae) .Biochem. Syst. Ecol., 19(7): 535-539.
- Lota ML, Serra D, Tomi F, Casanova J (2000). Chemical variability of peel and leaf essential oils of mandarins from Citrus reticulate Blanco. Biochem. Syst. Ecol., 28: 61-78.
- Lota ML, Serra D, Tomi F, Casanova J (2001). Chemical variability of peel and leaf essential oils of 15 species of mandarins. Biochem. Syst. Ecol., 29: 77-104.
- McLafferty FW, Stauffer DB (1991). The important peak index of the registry of mass spectral data.
- Salem A (2003). Extraction and identification of essential oil components of the peel, leaf and flower of tangerine"Citrus nobilis loureior var deliciosa swingle" cultivated at the north of Iran. Master of Science Thesis, Islamic Azad University-Pharmaceutical sciences Branch.

- Sawamura M, Minh Tu NT, Onishi Y, Ogawa E, Choi HS (2004). Characteristic odor components of citrus .reticulata Blanco (ponkan) cold pressed oil. Biosci.Biotechnol.Biochem., 68(8): 1690-1697.
- Sekiya J, Kajiwara T, Hatanaka A (1984). Seasonal changes in activities of enzymes responsible for the formation of C6-aldehydes and C6-alcohols in tea leaves, and the effects of environmental temperatures on the enzyme activities. Plant. Cell Physiol., 25(2): 269-280.
- Yajima I, Yanai T, Nakamura M, Sakakibara H, Hayashi K (1979). Composition of the volatiles of peel oil and juice from Citrus unshiu. Agric. Biol. Chem., 43: 259-264.
- Yoshikawa K, Kobayashi M, Arihara S (1996). Flower fragrance precursors from flower buds of Citrus Unshiu Marcov. Nat. Med., 50: 176-178.