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Volatile Differences of Pitted and Non-pitted 'Fallglo' Tangerine and White 'Marsh' Grapefruit

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Abstract. **Postharvest pitting, which has severely affected citrus quality, can be caused by wax application and high temperature storage. Internal volatile composition of waxed and non-waxed fruit could be an indicator of fruit susceptibility to postharvest pitting. In this study, volatile composition was compared between pitted and non-pitted 'Fallglo' tangerines [Bower citrus hybrid (*citrus reticulata* Blanco x *C. reticulata* Blanco x *C. paradisi* Macf.) x Temple (*C. reticulata* Blanco x *C. sinensis* L.)], as well as in white 'Marsh' grapefruit (*C. paradisi* Macf.). Pitted fruit had a higher volatile concentration than non-pitted 'Fallglo' tangerines or white 'Marsh' grapefruit. Concentrations of camphene, ethyl hexanoate, alpha-phellandrene, 3-carene, alpha-terpinene, p-cymene, and limonene were higher in pitted white 'Marsh' grapefruit than in those of non-pitted fruit. In 'Fallglo' tangerines, higher concentrations of limonene and citronellal were found in pitted fruits than in non-pitted fruit. In peel samples of grapefruit, seven different volatiles (methanol, ocimene, citronellyl acetate, alpha-copaene, *trans*-caryophyllene, alpha-humulene and valencene) were significantly higher in pitted peel than in non-pitted grapefruit peel. Volatiles, such as limonene could be used to predict peel disorders of white 'Marsh' grapefruit and 'Fallglo' tangerines during storage.**

Postharvest pitting of citrus is characterized primarily by oil gland breakdown in waxed fruit. In the advanced stage, peel tissue around the oil glands become dark brown, and then total peel collapse occurs (Petracek and Dou, 1998; Dou et al, 1999). Postharvest pitting is caused by wax application followed by high temperatures (>9 °C)during transit and storage, unlike the chilling injury which is caused by low temperature (<4.5 °C; Petracek and Dou, 1998). Petracek at el. (1998) found that shellac/resin based wax resulted in more severe pitting in grapefruit stored at 21 °C than carnauba or polyethylene waxes. Dou et al. (1999) found that resin/shellac solution causes pitting, but in lesser magnitude than resin/shellac wax. Storing waxed fruit at high temperatures increases fruit respiration rate. As a result, the fruit internal atmosphere changes due to stress associated physiology and biochemical processes. Theses changes are characterized by low O_2 and high CO_2 and ethonal partial pressures (Petracek et al., 1998). Also, wax limits fruit gas exchange (Hagenmaier and Show, 1992) and promotes anerobic respiration. Many volatiles will be produced due to the anaerobic respiration. The objective of this paper is to study the volatile differences between pitted and non-pitted whole fruit as well as peel samples in order to understand the development of fruit pitting.

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Material and Methods

Plant materials. White 'Marsh' grapefruit and 'Fallglo' tangerines were used in these experiments. These fruits were commercially harvested, and treated in a packinghouse and stored at 21 °C (70 °F) and 93% relative humidity (RH) for 14 d at the Citrus Research and Education Center, Lake Alfred, Fla. Fifteen fruits were randomly selected as a sample for the following studies.

Sample preparation of whole fruit. Two containers were packed each with previously selected 15 'Fallglo' tangerines or 'Marsh' grapefruit and sealed overnight, or at least for 12 h. The containers were sealed with silicon around the contact area and between the cover and container (GE Silkicon II; General Electric Co., Waterford, N.Y.). Volatile contents of the fruit and the atmosphere of the container were allowed to reach equilibrium during this period. The study contained two treatments (pitted vs. non-pitted fruit). Each treatment replicated four times.

Sample preparation of peel sample. Two grams of both albedo and flavedo with pitting symptoms were cut away using a razor at ≈0.2–0.3 cm^3 and put together in a 100-mL flask. The flask was covered with a rubber stopper and each fruit sample was allowed to reach equilibrium with the contained environment overnight. A treatment with a non-pitted peel was also set up in the same manner for the volatile analysis. Each treatment was replicated four times.

GC analysis. The whole fruit or peel samples for volatile analysis were collected by Solid Phase Micro Extraction (SPME) (Supelco, Bellefonte, Pa.) and injected into a Hewlett Packard 5890A gas chromatograph equipped with a 30 M × 0.32 mm RTX-5 column (Restec, Bellefonte, Pa.), hydrogen carrier gas and a FID detector. Data were analyzed using Chrom Perfect Spirit (Justice Laboratory Software, Denville, N.J.). SPME proved to be the most reliable method of collecting and analyzing the volatiles released by the fruit. The advantage of the SPME method is that it eliminates solvent effects and can capture more volatile components on a GC column than solvent extraction. The percentage of each volatile was recorded and stored in the data station.

Statistic analysis. The concentration of volatile components in pitted fruit or peel samples, respectively, vs. non-pitted fruit or peel samples were analyzed by analysis of variance (ANOVA) using Plotit software (Scientific Programming Enterprises, Haslett, Mich.). Duncan's mutiple range test was used to separate the means of the treatments at $P < 0.05$.

Results and Discussion

Differences in volatile constituents between pitted and non-pitted fruits. There are only a few chemicals which were found in significantly higher amounts in the pitted fruit than in the non-pitted fruit. These chemicals are different in grapefruit than in 'Fallglo' tangerines. Figs. 1 and 2 summarize the significant component percentages of pitted and non-pitted 'Fallglo' tangerines and white 'Marsh' grapefruit, respectively.

For 'Fallglo' tangerines, higher concentrations of limonene and citronellal were found in pitted fruit than in the non-pitted fruit. Limonene represents 58% of the total volatile components in pitted fruit and 41% in non-pitted fruit; showing that limonene content is significantly higher in pitted fruit (Fig. 1). Citronellal is another volatile component that was found to be in significantly higher amounts in pitted fruit than in non-pitted fruit. However, the magnitude of citronellal is much lower than limonene in packed 'Fallglo' tangerines. There were no significant differences between the pitted and non-pitted fruit regarding the other 120 volatile components analyzed (Data not shown).

Seven significantly higher concentrations of volatiles were found in pitted white 'Marsh' grapefruit than in those of the non-pitted fruit (Fig. 2). In most cases, these volatile concentrations were three to four times greater. For example, the content level of alpha-terpinene is 1.4% of the total volatile components of the atmosphere gases in non-pitted grapefruit and is 7.3% in pitted grapefruit. Limonene made up 10.8% of the contents in non-pitted fruit, whereas in pitted fruit, limonene made up 48.4%, which is about in line with the previously mentioned 'Fallglo' measurements. It has been found that limonene is present high amounts in a citrus peel (Singlair, 1972). Waxed fruit promotes anaerobic respiration; an environment that allows limonoids to be reduced to limonene. Logically, limonene is vulnerable to the fruit's internal atmosphere. Previous studies have indicated that pitting is correlated with higher internal ethanol and CO_2 and also with lower internal O_2 concentrations. Pitted fruit typically have a high concentration of ethanol (Petracek et al., 1998). In this study, limonene content was found to be higher in pitted fruit than in non-pitted fruit. This can be interpreted as a result of the progressive development of anerobic respiration due to wax application. In addition, the current studies reveal that the highest percentage of volatiles in fruit is limonene. Recently, Sun and Petracek (1999) reported that limonene levels in pitted fruit decrease at fruit oil glands compared to non-pitted fruit. The results from our experiments demonstrated that limonene is volatilized within the fruit's internal atmosphere. In addition, pitting increases with high temperature storage (Petracek and Dou, 1998). Sun and Petracek (1999) reported that limonene decreases within oil glands in increased storage temperatures. The results again confirm that decreased limonene in oil glands may be associated with high limonene concentration in the fruit internal atmosphere. In pitted fruit, the release of limonene from oil glands is greater than in that of non-pitted fruit (Figs. 1 and 2).

Differences in volatile constituents between pitted and non-pitted peel samples. In order to compare the differences in volatile components of pitted fruit and non-pitted fruit, the headspace of 2 g of pitted peel and two grams of non-pitted peel of white 'Marsh' grapefruit were measured with the GC. Seven volatile components were found to be significantly higher in pitted peels than in non-pitted peels (Fig. 3). For example, amount of *trans*-caryophyllene was almost double in pitted peel samples than in non-pitted peel samples. Seven volatile components measured in the peel samples were different from the components of the whole fruit. However, the total magnitude of these seven components of pitted and non-pitted peels are smaller than those measured in whole grapefruit (Figs. 2 and 3). In most cases, volatile components in peel samples are smaller in molecular weight than those in whole fruit. It is assumed that biochemical processes in peel samples are more intensive and inconsistent. This is due to exposure to aerobic respiration and wound response as a result of cutting the peel samples. Afterwards, peel samples were immediately set up in the flask for measurement. Volatiles found in peel samples were the same as those reported in volatile components of grapefruit juice (Shaw et al., 2000).

In summary, postharvest pitting is characterized by the breakdown of oil glands after waxing and storage at high temperatures (Dou et al., 1999; Petracek and Dou, 1998). In this study, the large amounts of volatiles found in pitted fruit and non-pitted fruit are essential oils such as limonene and valencene. It is speculated that limonene and valencene are both related to postharvest pitting. In addition, volatile measurements in whole fruit are more closely related to fresh fruit postharvest conditions than measurements using cut peel samples. Therefore, whole fruit volatiles can be related to the development of pitting and other quality characteristics. Limonene is the only volatile found in significantly higher amounts in pitted 'Fallglo' tangerines and 'Marsh' grapefruit as compared to non-pitted fruit. Chemical analysis of limonene can be a useful indicator of peel pitting.

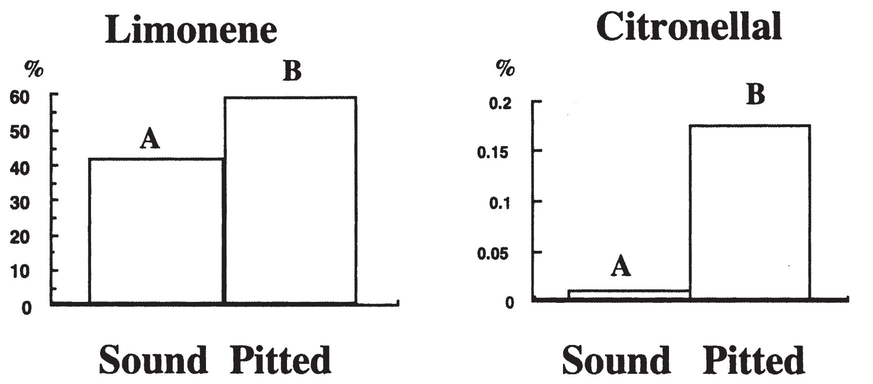

Fig. 1. Percentage of volatile concentrations that were significantly different between non-pitted and pitted whole 'Fallglo' tangerines. Different letters at each graphic represent the significant differences between the (**A**) non-pitted and (**B**) pitted fruit at $P \leq 0.05$.

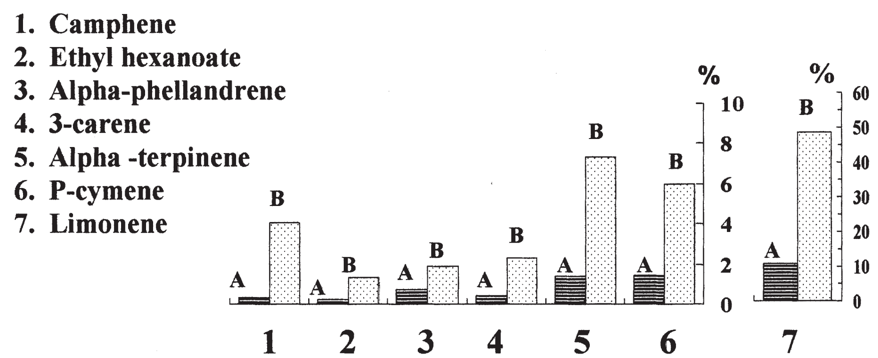

Fig. 2. Percentage of volatile concentrations that were significantly different between non-pitted and pitted whole grapefruit. Different letters at each volatile component represent the significant differences between the (**A**) non-pitted and (**B**) pitted fruit at $P \leq 0.05$.

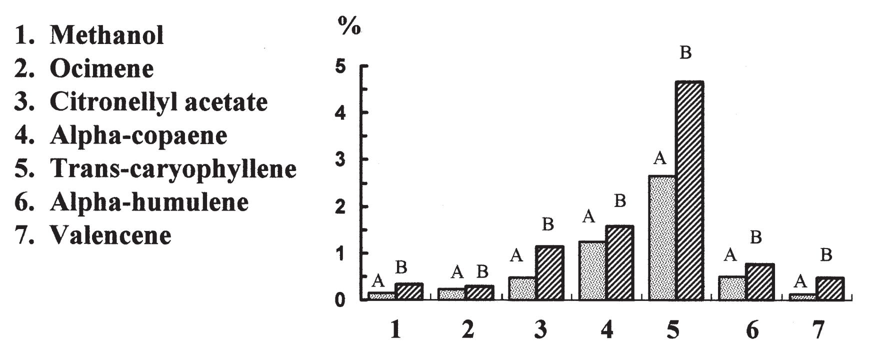

Fig. 3. Percentage of volatile concentrations that were significantly different between non-pitted and pitted peel of white 'Marsh' grapefruit. Different letters at each volatile component represent the significant differences between the (**A**) non-pitted and (**B**) pitted peel sample of grapefruit at $P \leq 0.05$.

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