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Stability of Headspace Volatiles in a 'Fallglo' Tangerine Juice Matrix System at Room Temperature

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Gas chromatography systems are usually equipped with autosamplers. Samples held in the autosampler tray can stay up to one day or longer at room temperature, if the tray is not equipped with a cooling mechanism. The objective of this research was to determine if holding samples at room temperature influences measurement of volatiles by using 'Fallglo' tangerine juice as a model. Tangerine juice mixed with saturated NaCl containing an internal standard (IS), 3-hexanone, was held in the autosampler for different periods of time at 25 °C, incubated at 40 °C, and then exposed to a solid phase microextraction (SPME) fiber to absorb volatiles at 75-min intervals prior to be analyzed by a GC-MS system. Results showed that there were significant changes caused by a 24 h or longer holding time at 25 °C in absolute peak area (APA) for 12 volatiles out of a total of 16 compounds detected, including hexanal, *E*-2-hexenal, ß-pinene, octanal, *d*-limonene, linalool, nonanal, butyl-2-methylpropionate, copaene, caryophyllene, and valencene and the IS, 3-hexanone. However, there were no differences for four terpenes: α-thujene, α-pinene, α-terpinene and *p*-cymene. Three aldehydes (hexanal, *E*-2-hexenal and octanal) increased linearly at a rate of 144%, 238%, and 127% per day, respectively. The proportion agrees with a Henry's law based model. Butyl 2-methylpropionate, the only ester detected, decreased in both absolute and relative amounts (relative to IS), indicating that esters may not be favorable compounds in competition for binding sites on the SPME fiber chosen. Terpenes and terpene alcohols increased in absolute amount but decreased in relative amount as holding time was extended. The research confirmed that extended holding time at room temperature markedly influenced the profile of volatile measurements, and the change in IS during holding period did not represent other volatile components.

Gas chromatograph (GC) systems are very frequently equipped with an autosampler. Samples held in the autosampler can stay up to one day or longer at room temperature. The prerequisites for headspace volatile research under such conditions are based on the following assumptions: there are no chemical and/or biochemical reactions in both the liquid and gas phases; changes for partitioning into the headspace at room temperature are negligible in comparison with 40 °C incubation; and all headspace concentration changes caused by holding the sample at room temperature is represented by changes in an internal standard (IS).

To prevent volatile changes caused by chemical, enzymatic, and microbiological reactions during storage of juice samples in the vials, various procedures are used in sample preparation. Pasteurization, a standard process for commercial production of orange juice, exposes the juice to high temperatures for a short time (such as 90 °C for 10 s) to inhibit microbial population growth and to inactivate enzyme activity (Kimball et al., 2004). Citrus juice is usually acidic with a pH lower than 4 (Hearn, 1987). Under such a low pH, most bacteria are inactivated, however, yeasts and fungi can tolerate a pH much below this, and cause spoilage of juice (Mahale et al., 2008). Low pH also inhibits activity of most enzymes (Perez and Sanz, 2008). For volatile analysis, juice is usually mixed with sodium chloride to form a juice-NaCl matrix (Baldwin et al., 1995) to control water activity (a_w) (Halling, 1992) as well as to inactivate enzymes. Table 1 shows a_w value of various NaCl solutions. Most bacteria do not grow at a_w below 0.91, and most molds cease to grow at a_w below 0.80 (Marianski and Marianski, 2008). The result of mixing juice with a saturated NaCl solution (≈36%) in a 50/50 dilution yields a concentration of ≈18% NaCl with an a_w of ≈0.89. Under such a low a_w value, growth of bacteria and activity of enzymes is greatly suppressed. Vial headspace also can be replaced by nitrogen or other inert gases to protect volatiles from oxidation in both liquid and gas phases. In this research we used all the protection methods described above except for pasteurization, to eliminate effects caused by chemical, enzymatic, or microbiological reaction.

Partition of volatile components from liquid to gas phase follows Henry's law (Sangster, 2003). Although incubation at 40 °C for 1 h, a setting often used by analysis of headspace volatile GC (Baldwin et al., 2010; Bazemore et al., 1999; Miyazaki et al., 2011), generates a significant amount of vapor in the headspace, our preliminary experiment showed that holding the sample for different lengths of time in the autosampler at room temperature prior to incubation at 40 °C markedly influenced the volatile profiles. The objective of this research was to determine how holding time in an autosampler at room temperature influences volatile profiles of fresh 'Fallglo' juice under a regular GC- mass spectrometer (MS) headspace analysis system.

Table 1. Water activity (A_w) of various NaCl solutions (Marianski and Marianski, 2008).

A_w	0.995	0.99	0.98	0.96	0.94	0.92	0.90	0.86
NaCl (%)	0.9	1.7	3.5	7.0	10.0	13.0	16.0	22.0

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Material and Methods

Plant materials and juice preparation. Fresh 'Fallglo' tangerines were purchased from a local grocery and then were hand juiced carefully using an Oster Model 3183 citrus juicer (Household Appliance Sales and Service, Niles, IL) on 15 Nov. 2010. The juice (50 mL) was mixed with 50 mL of a saturated NaCl solution and the internal standard (IS, 3-hexanone with a final concentration of 10 µM). The juice-NaCl matrix (6 mL) were pipetted into a 20 mL vial, the headspace was flushed with nitrogen gas, and then the vials were crimp capped with Teflon/silicone septa. Sample vials were stored at –20 °C until being thawed under tap water and loaded into the autosampler (Model MPS2, Gerstel Inc., Linthicum, MD).

Treatments. Identical tangerine juice-NaCl samples were held in the autosampler at 25 °C for 2.00, 3.25, 4.50, 5.75, 7.00, 8.25, 26.00, 27.25, 28.50, 29.75, 31.00, or 32.25 h (75-min intervals), respectively, prior to being incubated and analyzed.

Headspace sampling and GC-MS analysis. After holding for different time periods in the autosampler, juice samples were incubated for 30 min at 40 °C. A 2-cm solid phase microextraction (SPME) fiber (50/30 µm DVB/Carboxen/PDMS; Supelco, Bellefonte, PA) was then exposed to the headspace for 30 min at 40 °C. After exposure, the SPME fiber was inserted into the injector of a GC-MS (Model 6890, Agilent, Santa Clara, CA) to desorb the extract for 15 min at 250 °C. The GC-MS equipment and settings were: DB-5 (60-m length, 0.25-mm i.d., 1.00-µm film thickness; J&W Scientific, Folsom, CA) columns, coupled with a 5973 N MS detector (Agilent Technologies). The column oven was programmed to increase at 4 °C·min^{-1} from the initial 40 °C to 230 °C, then ramped at 100 °C·min^{-1} to 260 °C and held for 11.70 min for a total run time of 60 min. Helium was used as carrier gas at flow rate of 1.5 mL·min^{-1}. Inlet, ionizing source and transfer line were kept at 250, 230, and 280 °C, respectively. Mass units were monitored from 40 to 250 m/z and ionized at 70 eV. Data were collected using the ChemStation G1701 AA data system (Hewlett-Packard, Palo Alto, CA). A mixture of C-5 to C-18 n-alkanes was run at the beginning of each day to calculate retention indices (RIs).

Volatile compound identification. Volatile compounds were identified by comparison of their mass spectra with library entries (NIST/EPA/NIH Mass Spectral Library, version 2.0d; National Institute of Standards and Technology, Gaithersburg, MA), as well as by comparing RIs with published RIs (Adams, 2007; Kondjoyan and Berdagué, 1996).

Absolute and relative peak area (APA and RPA). Amount of each volatile compound was represented as APA of total ion current (TIC). The RPAs of individual volatiles were calculated by dividing the APA with IS APA, i.e., RPA = APA / (IS APA).

Statistical analyses. SAS Version 9.1 (SAS Institute, Cary, NC) was used for analysis of volatile data. Each component was analyzed using *t*-test (PROC TTEST) to compare how a 24-h longer holding time (2–8.25 h vs. 26–32.25 h) at 25 °C affect APA and RPA. Linear regressions (PROC REG) of APA or RPA vs. holding time were calculated and used to predict increase or decrease per day for each component.

Results

Fifteen compounds, including nine terpenes (α-thujene, α-pinene, ß-pinene, α-terpinene, *p*-cymene, *d*-limonene, copaene, caryophyllene, and valencene), one terpene alcohol (linalool), four aldehydes (hexanal, (*E*)-2-hexenal, octanal and nonanal), and one ester (butyl 2-methylpropionate) were identified (Table 2). By comparison with 2- to 8.25-h holding time in the autosampler, an average 24 h longer holding time at 25 °C increased the APA by 45% for the IS, 28.5% to 139.0% for aldehydes and 23.1% to 33.1% for five terpenes; however, there were no significant changes for the other four terpenes (Table 2). The terpene alcohol, linalool increased 14.5%, but the ester, butyl 2-methylpropionate decreased 38.7% as holding time increased (Table 2). The total APA increased 30.4% (Table 2). Twelve out

Table 2. Changes of peak area of volatiles as holding time was extended for 24 h longer in autosampler at 25 °C.[z]

Compound	Retention time (min)	RI	Absolute peak area (APA) increased/decreased (%)	Relative peak area (RPA) increased/decreased (%)
3-Hexanone (internal standard)	15.0	784	45.0***	---
Hexanal	15.7	803	98.8***	36.0**
(*E*)-2-Hexanal	18.2	862	139.0***	71.0***
α-Thujene	21.8	943	–35.3 NS	–57.0**
α–Pinene	22.4	955	21.5 NS	–15.4 NS
ß-Pinene	24.4	1000	33.1*	–6.6 NS
Octanal	25.0	1013	88.9***	29.9*
α-Terpinene	26.0	1035	16.1 NS	–21.7 NS
p-Cymene	26.3	1043	8.1 NS	–27.6**
d-Limonene	26.6	1048	23.1*	–15.1*
Linalool	29.1	1106	14.5*	–21.7***
Nonanal	29.3	1110	28.5**	–12.1 NS
Butyl 2-methylpropionate	39.9	1382	–38.7**	–59.5***
Copaene	40.5	1400	31.1**	–8.0 NS
Caryophyllene	42.2	1455	32.7**	–7.1 NS
Valencene	44.5	1532	29.6**	–12.2*
Volatile abundance			30.4**	–14.9*

[z]The absolute and relative peak areas in samples held for 26 to 32.25 h were compared with the areas held for 2 to 8.25 h.
NS, ***, **, *Nonsignificant or significant at $P < 0.001$, 0.01, and 0.05, respectively.

of the total 16 compounds (75%) showed significant increase or decrease (Table 2). On the other hand, changes of the RPA showed different patterns. Three out of the four aldehydes significantly increased, however, nonanal, the aldehyde with the largest molecular weight did not significantly change (Table 2). All nine terpenes decreased, but only four were statistically significant (Table 2). Both linalool and butyl 2-methylpropionate decreased significantly (Table 2). The total volatile abundance significantly increased by absolute value, and conversely, decreased by relative value (Table 2).

Changes of individual volatiles over the holding period are shown in Fig. 1. APA of three aldehydes (hexanal, *E*-2-hexenal and octanal) increased linearly (level of significance of $r^2 = 0.05$), at a rate of 144%, 238%, and 127% per day, respectively (Table 3). The results can also be expressed as a doubling of APA within 10.1 to 18.9 h. Other components did show increases (Table 2), but not in a linear manner (Table 3 and Fig. 1). For relative values, (*E*)-2-hexenal increased linearly with a rate of 86.2% per day, but butyl 2-methylpropionate decreased linearly (–53.2% per day) (Table 3). Other components did not show change in a linear manner (Table 3).

Discussion

Response of volatile peak areas to holding time can be categorized into three types: (1) the APA markedly increased with a linear manner and the relative values (relative to IS) also increased, such as hexanal, (*E*)-2-hexenal, and octanal; (2) both APA and RPA decreased, such as α-thujene and butyl 2-methylpropionate; and (3) the APA increased with a small margin, however the RPA decreased, such as happened with most terpenes and terpene alcohols (Tables 2 and 3, and Fig. 1).

Henry's law and partition of volatiles. Henry's law is one of the gas laws. It is defined as: "at a constant temperature, the amount of a given gas that dissolves in a given type and volume of liquid is directly proportional to the partial pressure of that gas in equilibrium with that liquid" (Wikipedia, 2011). An equivalent way of stating the law is that the solubility of a gas in a liquid at a particular temperature is proportional to the pressure of that gas above the liquid. Henry's law has been shown to apply for a wide range of dilute solutions (Sangster, 2003). Fresh orange juice contains more than 99.5% aqueous solution (Bai et al., 2010) and oxygenated compounds, such as low molecular weight esters and aldehydes, are mainly contained in the juice serum (Bai et al., 2010; Brat et al., 2003; Radford et al., 1974). In this section, we will discuss hexanal, (*E*)-2-hexenal and octanal, all of which showed substantial increases in a linear manner with increased holding time at room temperature, and are very soluble in juice serum. Another soluble compound, butyl 2-methylpropionate should also exhibit partitioning into the headspace following Henry's law. However, the measured peak areas were strongly influenced by the selective absorption of the SPME, which will be discussed in the next section.

Henry's law constant, k_H, is calculated according to

$$k_H = c/p \quad (1)$$

where c is concentration of the solute in the solution (mol/L) and p is pressure of the solute in the gas above the solution (bar).

k_H value at the standard condition (298 °K) is expressed as k^{θ}_H. The k_H value at a given temperature T is expressed as $k_H(T)$. It changes along with temperature change.

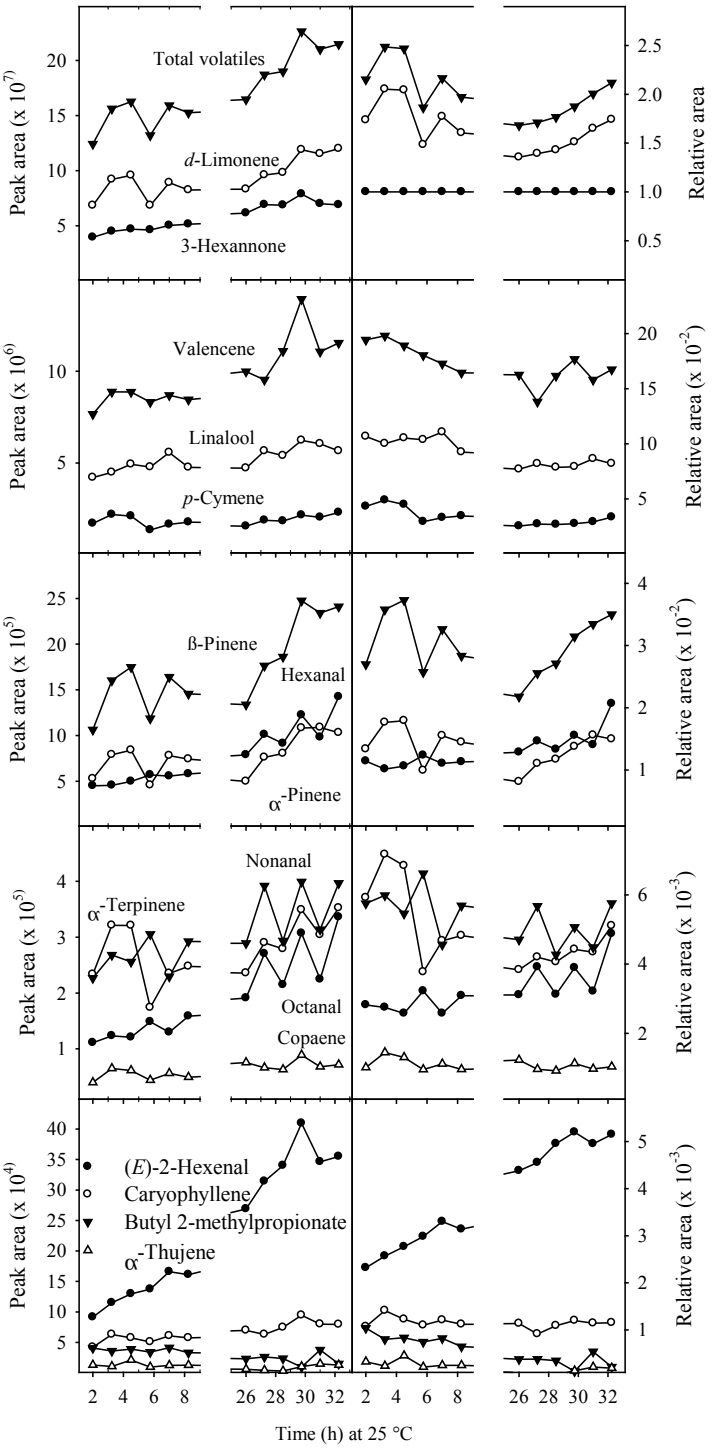

Fig. 1. Changes of absolute (total ion current, **left**) and relative (**right**) peak areas of volatile compounds, as influenced by extended holding time of samples in autosampler. Tangerine juice samples were incubated at 40 °C for 30 min prior to exposing to a SPME fiber 30 min.

Thus, the formulation is

$$k_H(T) = k^{\theta}_H \cdot \exp[C(1/T - 1/T^{\theta}] \quad (2)$$

where *C* is temperature dependence constant, T and T^{θ} are the thermodynamic and the standard condition temperature (absolute, °K), respectively.

Table 3. Estimated increase or decrease of absolute and relative peak areas (APA and RPA, respectively) caused by extending holding time of samples in autosampler at 25 °C.[z]

Compound	Rate of increase or decrease of APA (%/d)	Rate of increase or decrease of RPA (%/d)
3-Hexanone (internal standard)	56.5	---
Hexanal	144.1*	42.1
(*E*)-2-Hexanal	238.6*	86.2*
α-Thujene	–28.5	–49.1
α–Pinene	35.3	–12.4
ß-Pinene	50.1	–4.3
Octanal	126.7*	34.3
α-Terpinene	19.6	–21.3
p-Cymene	10.4	–26.4
d-Limonene	31.9	–13.9
Linalool	19.9	–20.2
Nonanal	36.0	–11.5
Butyl 2-methylpropionate	–36.6	–53.2*
Copaene	39.4	–9.2
Caryophyllene	44.8	–6.5
Valencene	35.5	–12.1
Volatile abundance	39.6	–13.8

[z]Linear regression for each compound was calculated based on APA and RPA over 12 holding times in 32.5 h. Internal standard was 3-hexanone.
*Indicates significance of r^2 at 0.05 level.

Among the 15 volatile compounds detected in 'Fallglo' tangerine juice, there were 6 compounds that had published Henry's law constants according to NIST Chemistry WebBook (Linstrom and Mallard, 2011), in which only three compounds had published temperature dependence constants (Table 4) (Linstrom and Mallard, 2011).

Take hexanal as an example. Under our condition of analysis, 6 mL of juice-NaCl matrix was in the 20-mL vial, leaving 14 mL as headspace. Hexanal concentration in the headspace would be 1.91% and 5.00% of total hexanal in the system at equilibrium status at 25 °C and 40 °C, respectively (Table 4). Assuming k_H value is proportional to the partition rate for the compound, the partition rate at 40 °C is 2.62 times faster than at 25 °C. It means that the partition caused by a 24 h holding at 25 °C is equivalent to that caused by a 9-h incubation at 40 °C. It explains why the extended holding at room temperature is not irrelevant, and why measurements of hexanal, (*E*)-2-hexenal, octanal were markedly increased by extended holding time at 25 °C.

As a comparison, if the autosampler was cooled to 5 °C, hexanal concentration in the headspace would be 0.43% of the total content at equilibrium status. It would be 11.63 times smaller than at 40 °C, indicating a cooled autosampler would help in eliminating the influence caused by the pre-incubation holding times.

SPME extraction. SPME, is a sample extraction technique. It has been extensively applied to the study of orange juice aroma (Bazemore et al., 1999; Miyazaki et al., 2011; Rega et al., 2003). An ideal SPME should absorb all analytes non-selectively, however, many reports showed that SPME is selective and the capacity is not unlimited (Bazemore et al., 1999; Rega et al., 2003; Yang and Peppard, 1994).

Peak area of butyl 2-methylpropionate and α-thujene decreased by 38.7% and 35.3%, respectively when all other compounds increased with a total increase of 30.4% in APA (Table 2). The decrease of RPA of butyl methylpropionate exhibited a linear trend (Table 3). These results suggest that there is a binding-site competition mechanism. When there are less of the total volatile molecules in the headspace system, butyl 2-methylpropionate molecules can bind to the SPME; however, as total volatile molecules increase, butyl 2-methylpropionate molecules have less chance to be bound (Fig. 2).

Influence caused by peel oil and pulp in the juice. Citrus juice contains 0.3% to 0.5% insoluble solids (pulp) and 0.01-0.03% peel oil (Bai et al., 2010). For the volatile compounds that strongly interact with the pulp and peel oil, the partition would not simply follow the Henry's law. Studies on the distribution of volatile compounds between pulp and serum in different fruit juices showed that hydrocarbons (mono and sesquiterpene hydrocarbons) were associated with pulp (Brat et al., 2003; Radford et al., 1974). By separating pulp from juice using centrifugation, these compounds greatly decreased in the serum (Jordan et al., 2001). Volatile compounds associated with peel oil include limonene, myrcene, α-pinene, octanal, nonanal, decanal, sinensal, and linalool (Shaw, 1991). The partition of those pulp and peel oil associated compounds is complicated. Fig. 3 is a hypothetical model which shows how the peel oil associated compounds equilibrate with micro-oil drops, then serum and finally into the headspace in a multi-equilibration system, and similarly, pulp associated compounds similarly equilibrate with pulp particles, then serum and finally headspace in a multi-equilibration system. There would be a direct partitioning from micro-oil drops or pulp particles to headspace on the juice surface, which would also be negligible in a cool and static system, however, this would become more important in a hot and dynamic (agitating) system.

Conclusion

There is a risk when analyzing headspace volatile concentration by GC equipped with an autosampler at room temperature, especially if the holding time is extensive. Generally, APA increased as holding time increased, and RPA decreased at same

Table 4. Effect of temperature on Henry's law constant and distribution of selected 'Fallglo' juice volatile aldehydes in the gas phase at equilibrium status.[z]

Compound	Molecular wt	k_H(mol·kg^{-1}·bar^{-1})				Distribution in gas phase (%)[y]		
		k^{θ}_H	k_H(278 °K)	k_H(313 °K)	*C*	298 °K (25 °C)	278 °K (5 °C)	313 °K (40 °C)
Hexanal	100	4.9	23.50	1.72	6500	1.91	0.43	5.00
Octanal	128	2.1	12.51	0.64	7400	4.35	0.81	12.44
Nonanal	142	1	5.03	0.34	6700	8.71	1.99	21.05

[z]k_H(T) and k^{θ}_H: Henry's law constant at thermal-dynamic and standard condition (298 °K), respectively; *C*: temperature dependence constant.
[y]20-mL vial containing 6 mL juice-NaCl matrix and 14 mL headspace.

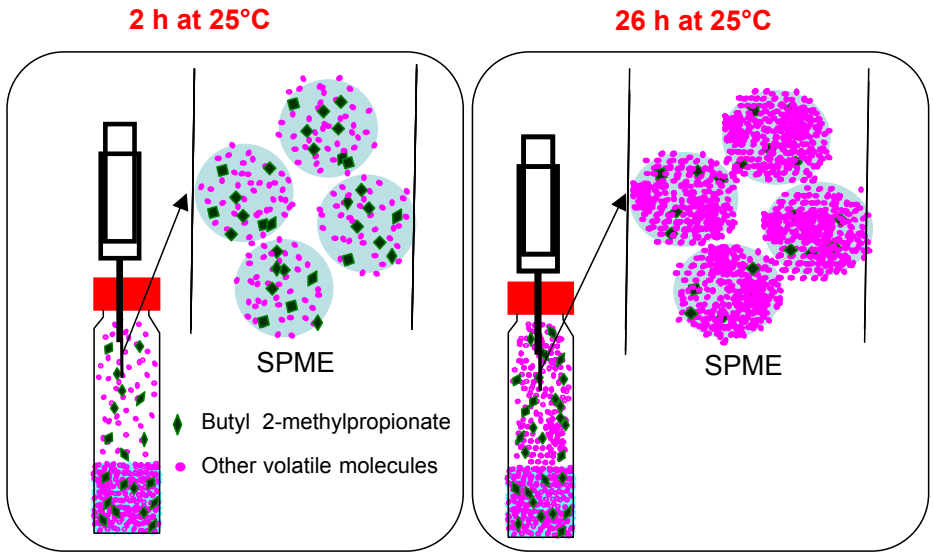

Fig. 2. Hypothetical model for the competition of volatiles for the binding sites on the SPME. **Left panel** shows when there are less of the total volatile molecules in the headspace system, butyl 2-methylpropionate molecules have been bound; however, as total volatile molecules increasing, butyl 2-methylpropionate molecules have less chance to be bound (**right panel**).

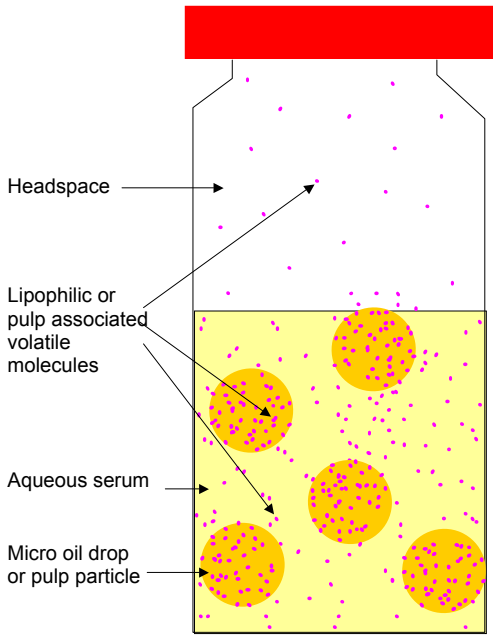

Fig. 3. Hypothetical model for the partition of volatile molecules associated with peel oil or pulp in a micro-oil drops or pulp particles–aqueous serum–headspace multi-equilibrate system. There are directive partitions between micro-oil drops or pulp particles vs. headspace in the juice surface.

time. However there were exceptions, in which both APA and RPA decreased such as α-thujene, and butyl 2-methylpropionate, and both APA and RPA increased such as with three aldehydes. Peak areas measured by GC-TIC showed that partitioning of aqueous aldehydes and ester compounds between juice and headspace followed Henry's law; selective binding of SPME influences volatile profile for ester and some terpenes; and partition of peel oil and pulp associated volatiles from juice to headspace follows a complex multi-equilibration system.

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