Abused Thermal Treatment Impact on Degradation Products of Chilled Pasteurized Orange Juice

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Kinetic analyses of data generated on the degradation products of fresh-chilled, pasteurized orange juice submitted to severe heating time and temperature protocols (75, 85, and 95 °C for 15, 30, and 60 min) are presented. The influence of pH (3.1, 3.8, and 4.5) and oxygen content (deaerated and oxygenated) on 2-hexanal, furfural, α-terpineol, *p*-vinylguaiacol, and other degradation byproducts was studied using gas chromatography/mass spectrometry. The effects of elevated temperature protocols on the generation of *p*-vinylguaiacol and α-terpineol were demonstrated. The reaction mechanisms are pseudo-zero-order. The study on the influence of pH indicated that the acid-catalyzed hydration−dehydration pathway (ACHD) was the main reaction yielding *p*-vinylguaiacol and α-terpineol. *d*-Limonene was lost to only a minor extent through one of the two competing pathways, the well-known ACHD pathway, as none of the typical oxidation byproducts were detected in either the deoxygenated or aerated orange juice in which *d*-limonene was deliberately added.

Keywords: *Kinetics; time−temperature effects; chilled pasteurized orange juice; citrus beverage degradation*

INTRODUCTION

Citrus juices are harsh environments that expose juice components and flavors to both acidic conditions and dissolved oxygen. Changes occurring in the citrus juices after processing and subsequent storage result in the loss of the original flavor and the development of detrimental off-flavor components. Numerous degradation reactions occur at the same time, and each of these reactions may influence the course of other reactions. Thermal processing operations (Nagy et al., 1989; Rassis and Saguy, 1995) and prolonged storage (Moshonas and Shaw, 1989) promote these reactions.

The deterioration of orange juice quality results from a complex series of chemical reactions with differing reaction rates involving substrates such as essential oils, phenolic acids, sugars, lipids, ascorbic acid, and sulfur-containing compounds. Work done on the subject indicates that more than one type of mechanism is involved in the development of off-flavors. Off-flavors have been thought to originate primarily from peel oil constituents and juice solids. *d*-Limonene, the principal component of peel oil, undergoes a series of oxidative hydration−dehydration reactions (ACHD pathway) under acidic conditions, producing hydroxy compounds such as α-terpineol, cineol, and other products (Blair et al., 1952). Due to its unsaturated character, *d*-limonene can also yield byproducts such as carvone, carveol, 2,8-menthadiol, and various other compounds via radical formation by an oxidative pathway. The addition of *d*-limonene and linalool to a model juice resulted in a decrease of both compounds over storage, and this reaction was temperature dependent (Askar et al., 1973a,b). Both compounds were degraded to

α-terpineol and *cis*-1,8-*p*-menthanediol, while linalool degraded to nerol and geraniol. Hexanal is often selected as a criterion to estimate the extent of lipid and carotenoid oxidation (Lund and Bryan, 1977). Generation of *n*-octanal and *n*-decanal during storage as a result of the degradation of free, long-chain, unsaturated fatty acids has also been reported (Askar et al., 1973a,b). Degradation of various phenolic compounds produced undesirable off-flavor compounds such as *p*-vinylguaiacol, which imparts an old fruit and rotten flavor to orange juice at levels of 0.75 ppm, its concentration being more pronounced with higher juice content. This degradation is derived from phenolics and phenolic acids and is generated by the thermal decarboxylation of ferulic acid, which is found mostly in bound forms (glycosides, esters, and amides) in juice solids. Acidity, thermal processing, and subsequent high-temperature storage provide ideal conditions for the release of ferulic acid from its bound forms (Naim et al., 1988; Sutherland et al., 1995; Lee and Nagy, 1990; Peleg et al., 1992).

Most of the degradation reactions have been found to be first order (Lund, 1975); that is, the rate of formation or degradation of the component is directly proportional to its concentration. In food systems, there are usually sufficient concentrations of reactants for the chemical reactions during storage. Additionally, the concentration of reactants remains relatively constant over time (Toledo, 1980), implying that the rate of formation of degradation product is also constant with time. Furthermore, the temperature dependence of chemical reaction rates in biological systems can be expressed using an Arrhenius equation or in terms of Q_{10}, which is defined as the change in reaction rate constant accompanying a 10 °C change in temperature (Cohen et al., 1987).

Many chemical tests have been suggested as an indication of overall change in citrus juice. Furfural,

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which stems from the decomposition of sugar, amino acid-reducing sugar condensation, and ascorbic acid degradation, has been strictly regarded as an index of product history and shelf life since it is well established that the concentration of this compound increases over the incubation time of juice-based products, although it does not contribute to off-flavor (Nagy et al., 1972; Nagy and Dinsmore, 1974; Lee and Nagy, 1996). The strong relationship between the loss of vitamin C and the increase of furfural via an oxidative pathway has been often reported (Nagy and Dinsmore, 1974; Shaw et al., 1993). Vitamin C breakdown and furfural buildup follow a pseudo-zero-order reaction (Cohen et al., 1987) as a function of storage time at 4, 22.5, 35, and 45 °C (Kaanane et al., 1988).

Most of the publications on the development of off-flavor components in citrus products deal with prolonged storage periods of juice based-products packed aseptically at 96 °C for 16 s and then stored at ambient temperatures for several months (Shaw et al., 1993). There are few data available on the effect of severe thermal protocols as a means of predicting the kinetic of degradation products and disappearance of the precursors.

The objective of this work was to assess an abused time/temperature protocol for predicting the development of degradation products in an orange juice-based product and to determine the influence of factors such as oxygen and pH on the behavior of the precursors and their degradation products.

MATERIALS AND METHODS

Solvent Extraction Procedure. Pasteurized Tropicana orange juice (12 ± 0.4°Brix) from Florida was collected from a gable-top container under refrigerated conditions at a local supermarket. Five grams of NaCl was added to 10.0 mL of orange juice in a 15 mm × 15 mm culture tube with a Teflon-lined screw cap. The juice was spiked with 10 μL of internal standard (94 mg/mL D4cyclohexanone and 94 mg/mL D5phenol, Cambridge Isotope Laboratories). Samples were capped and shaken vigorously for 10 s. One milliliter of ethyl ether was added, and tubes were shaken vigorously for 50 s and then centrifuged for 2 min at 1500 rpm. The tubes were shaken for another 50 s and centrifuged for 5 min at 1500 rpm. Approximately 250 μL of the supernatant was transferred to an autosampler vial containing reduced volume insert (300 μL) and capped immediately.

Gas Chromatographic Analysis. Analyses were performed using a Finnigan Mat Incos 50 GC/MS (Thermo Instruments, San Jose, CA) scanned from 35 to 250 amu at 0.43 scans/s for 31 min. The GC column employed was a 0.25 mm × 30 m, 0.25 μm film, D-Wax (J&W Scientific, Folsom, CA), and the injection port and transfer line were maintained at 250 °C. Oven temperature was programmed from 40 to 90 °C at 3 °C/min and held for 1 min. The oven was then programmed at a rate of 10 °C/min to 120 °C and held for 2 min; this was then followed by a third ramp of 20 °C/min to 200 °C and held for 5 min. A 1 μL spit injection (50:1, flow rate = 1 mL/min helium) was performed using a CTC autosampler.

A standard curve containing four points varying in concentration by an order of magnitude was plotted before each set of samples was tested. Standard concentrations were carefully chosen to bracket actual concentrations found in real samples and an R^2 of 0.999 or better was recorded for all compounds. Figure 1 illustrates baseline resolution for all compounds and internal standards. Component identification was performed by correlating retention time with mass spectrum matching using authentic standards obtained from Aldrich Chemical Co. (Milwaukee, WI) (see Table 1). All analyses were performed in triplicate, and results are reported as averages (μg/mL).

Orange Juice Treatments. *Thermal Treatments.* Ten milliliter aliquots of fresh pasteurized orange juice were immediately transferred to 15 cm × 1.5 cm culture tubes and capped with Teflon-lined caps. The tubes were then subjected to thermal treatment by immersion in a constant-temperature bath for 15, 30, and 60 min at 75, 85, and 95 °C. Once the thermal treatments were completed, the tubes were immediately cooled in an ice−water bath to halt the reactions. All treatments were performed in duplicate, and results are reported as averages.

Effect of pH. Ten milliliter aliquots of the fresh juice (pH 3.8) were adjusted to pH 3.1 and 4.5 with HCl and NaOH (0.1 N), respectively. Samples were heated to and held at 95 °C for 15 min and then analyzed by GC/MS as previously described. All experiments were performed in duplicate, and results are reported as averages.

Effect of Oxygen. The orange juice was stripped of volatiles by stirring under vacuum for 2 h followed by sparging with argon gas using a fritted glass bubbler for 15 min until no residual volatiles were detected by GC/MS. The control juice sample was then exposed to air and shaken vigorously to redissolve oxygen, while the deoxygenated juice was stored under an argon atmosphere in a glovebox. Aliquots of the fresh and deoxygenated juice were analyzed using a YSI model 55/12 dissolved oxygen meter (Scientific Division, Yellow Springs Instrument Co., Yellow Springs, OH). The aerated and deoxygenated orange juices dissolved oxygen levels were 6.20 and 0.60 mg/L, respectively, at ambient temperature. The stripped juice was spiked with 84, 210, or 420 μg of d-limonene, then heated at 95 °C for 60 min, and analyzed using the same procedure as previously described. All experiments were performed in duplicate. Results are reported as averages.

RESULTS AND DISCUSSION

Effect of Time−Temperature Treatments on Degradation Compounds. Results are reported as a difference between the actual concentration and the initial concentration ($C − C_0$). Chemical analysis of degradation compounds showed that furfural was detectable only at low levels when the orange juice was exposed to the more severe thermal conditions (results not shown). Contrary to observations on the behavior of furfural (which is known to exhibit gradual increase over long storage time in citrus juices), these data suggest that furfural would not serve as a good indicator to predict the stability of chilled pasteurized orange juice exposed to heat treatment of only 60 min at a minimum temperature of 95 °C (Nagy et al., 1972; Lee and Nagy, 1996; Marcy and Rouseff, 1984).

As seen in Figure 2, the concentration of 2-hexenal (initially at 0.014−0.015 μg/mL) as a function of time is almost linear at the lower temperature, that is, 75 °C. Higher temperatures promoted the generation of this compound, but a plateau at higher temperature was reached after a 15 min thermal treatment, irrespective of the temperature conditions. These trends may be explained by substrate limitations (lipids, carotenoids, etc.), oxygen limitation, and high antioxidant activity as overriding factors controlling the production of this compound.

The concentration of α-terpineol (1.903−2.580 μg/mL) found initially in the untreated chilled orange juice was close to the threshold limit of 2 ppm for the sensory detection of the stale, piney, and musty note (Tatum et al., 1975). Figure 3 revealed a pronounced increase in the α-terpineol level, which is related to the time−temperature treatment condition observed. The level of α-terpineol significantly exceeds the sensory threshold level at 85 and 95 °C. A linear relationship was found between the concentration ratio of α-terpineol and
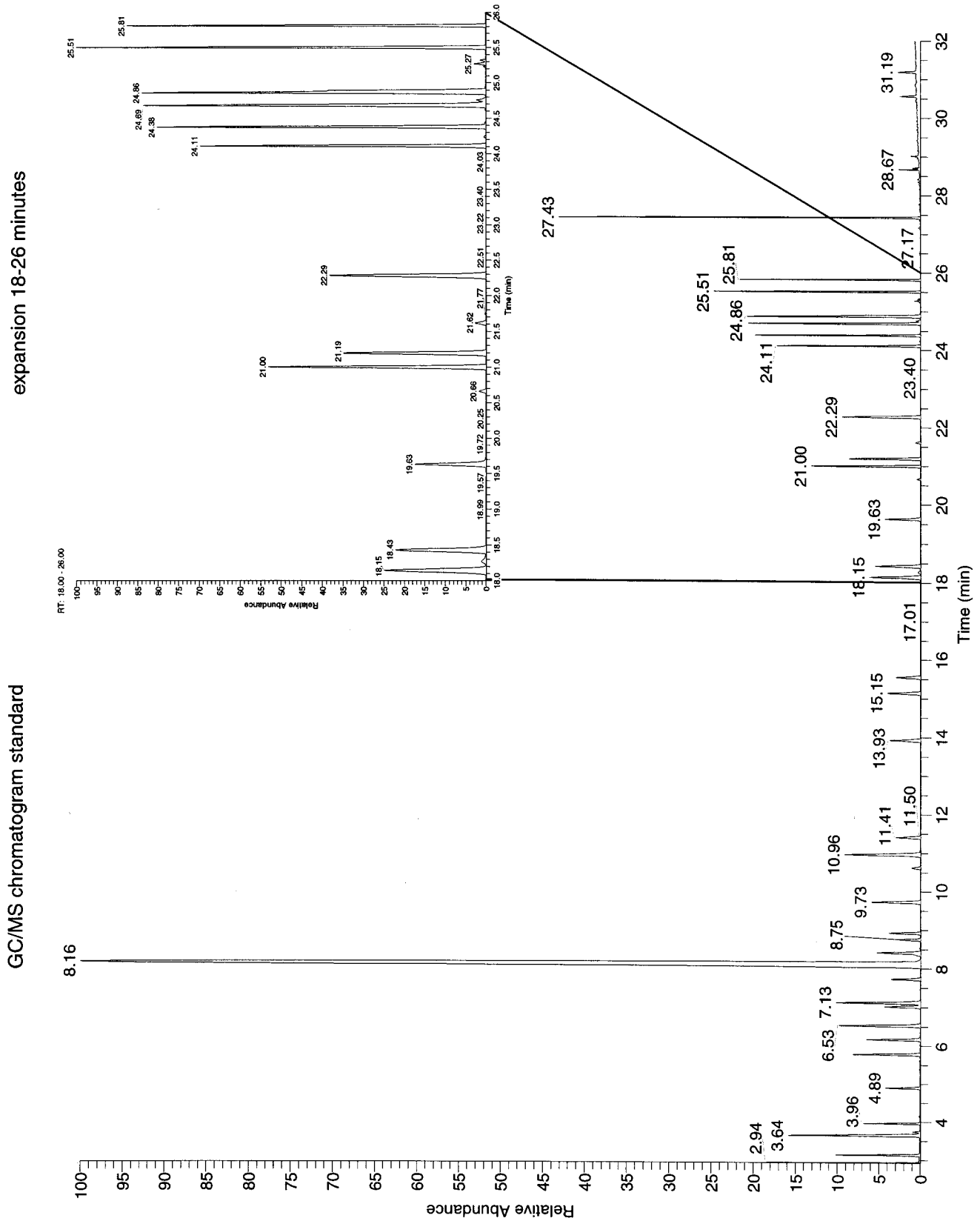
Degradation Kinetic of Chilled Pasteurized Orange Juice

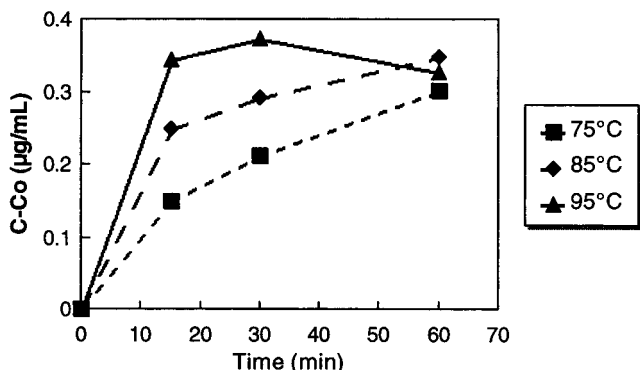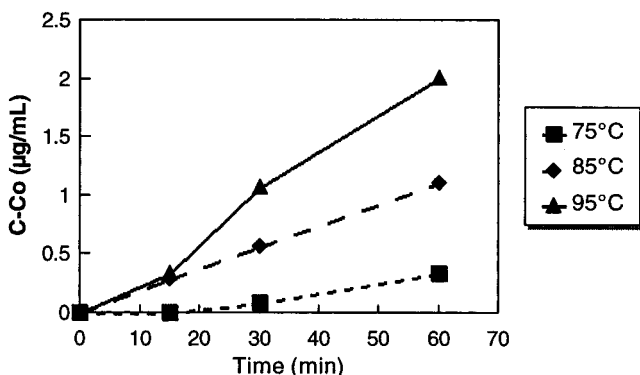*J. Agric. Food Chem.,* Vol. 46, No. 5, 1998 **1993**

Figure 1. GC/MS chromatogram of standards.

Table 1. Compound Retention Times

compound	retention time (min)	internal standard
D4cyclohexanone	10.96	*
D5phenol	27.43	*
hexanal	4.89	
2-hexenal	8.75	
α-terpineol	24.38	
p-vinylguaiacol	28.67	
furfural	18.43	
linalool	24.10	
valencene	24.67	

Figure 2. Concentration of 2-hexenal as a function of time.

Figure 3. Concentration of α-terpineol as a function of time.

the time of the thermal treatment at the three temperatures employed. This indicates that a pseudo-zero-order reaction would explain the behavior of this compound as a function of time.

Valencene and linalool, two flavor components found in orange juice, remained relatively constant with time under all time–temperature combinations used in this experiment. The evolution of p-vinylguaiacol (≈0.014–0.018 μg/mL initially) as a function of time in Figure 4 also indicates that production of this compound is a pseudo-zero-order reaction. It should be noted that 4-hydroxy-2,5-dimethyl-3(2H)-furanone, another major off-flavor, could not be detected at low levels due to the thermal instability of this compound (Walsh et al., (1997). Only the highest temperature condition, that is, 95 °C, promoted the formation of p-vinylguaiacol. The measured concentrations of p-vinylguaiacol in chilled orange juice (non thermally processed) were ≈5 times lower than the sensory threshold detection or 0.05 ppm (Tatum et al., 1975); however, the threshold value was significantly exceeded at 95 °C.

By applying linear regression analyses on the concentration ($C - C_0$) versus time data, the rate constants of 2-hexenal, α-terpineol, and p-vinylguaiacol were calculated as the slope of the regression that was forced through 0. For 2-hexenal, only the linear portion of the
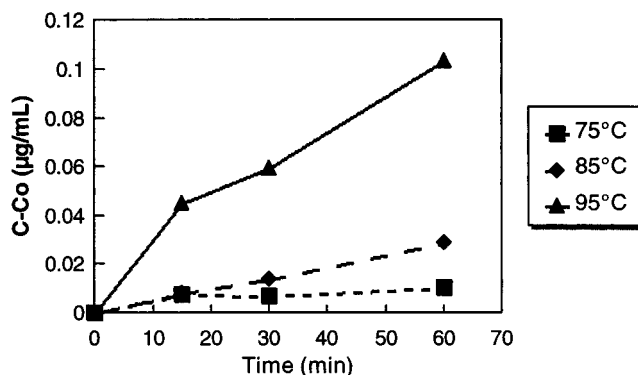
Figure 4. Concentration of p-vinylguaiacol as a function of time.

curve was used at high temperature (95 °C). The chemical reaction was assumed to be pseudo-zero-order. It has to be pointed out that Kaanane et al. (1988) also assumed a pseudo-zero-order reaction for reducing sugars and furfural, a degradation product. Moreover, in thermal processing, it is a common practice to assume a pseudo-zero-order reaction for degradation products as the concentration of reactants in food systems is usually high enough to remain constant (Toledo, 1980). The chemistry of degradation reactions is extremely complex; therefore, it is virtually impossible to identify the exact substrates and their concentrations. Values are reported in Table 2 in micrograms per milliliter per minute. The reaction kinetics of 2-hexenal are similar to those of α-terpineol. However, p-vinylguaiacol exhibited 10 times lower rate constants. Furthermore, the lower regression coefficient for 2-hexenal at high temperature may be attributed to substrate limitation and to possible analytical errors at low levels for p-vinylguaiacol at 75 °C. For 2-hexenal, a pseudo-zero-order reaction assumption is not valid. The low rate of formation of p-vinylguaiacol at low temperature as a function of time suggests that the extent of formation of this phenolic compound is minimal with a 12 °Brix orange juice treated for 60 min.

Activation energies were calculated from the logarithmic form of the Arrhenius equation:

$$\ln k = \ln \alpha + (-E_a/RT)$$

The natural log of rate constants was plotted against the reciprocal of the temperature, giving a constant negative slope which is equal to $-E_a/R$. Arrhenius plots for 2-hexenal, α-terpineol, and p-vinylguaiacol are shown in Figure 5. To obtain the activation energy (E_a) in calories per mole, the slope was multiplied by minus the value of R, the gas constant, which is 1.98 cal/mol·K. A good linear relationship was found for the Arrhenius plot for the three reactions (Table 3). An activation energy of 14.8 kcal/mol was found for 2-hexenal. Higher energy values of 20.2 and 28.3 kcal/mol for α-terpineol and p-vinylguaiacol, respectively, confirm that the deterioration mechanisms behave differently from that for 2-hexenal. All activation energies were within the order of magnitude of values reported for other quality parameters (Lund, 1975). Values of Q_{10} were calculated as a ratio of rate constants from 75 to 85 °C and from 85 to 95 °C for 2-hexenal, α-terpineol, and p-vinylguaiacol (Table 3). For chemical reactions, values of Q_{10} are usually on the order of 2 (Toledo, 1980), which is also observed in this experiment. As anticipated, the Q_{10}

Table 2. Rate Constants for the Degradation Reactions in Thermally Abused Orange Juice

	75 °C		85 °C		95 °C	
	k (μg/mL·min)	R^2	k (μg/mL·min)	R^2	k (μg/mL·min)	R^2
2-hexenal	0.565E−02	0.8472	1.111E−02	0.5378	1.810E−02	0.7675[a]
α-terpineol	0.480E−02	0.8553	1.860E−02	0.9997	3.330E−02	0.9870
p-vinylguaiacol	0.198E−03	0.5996	0.475E−03	0.9980	1.834E−03	0.9327

[a] Regression obtained in the linear portion of the curve.
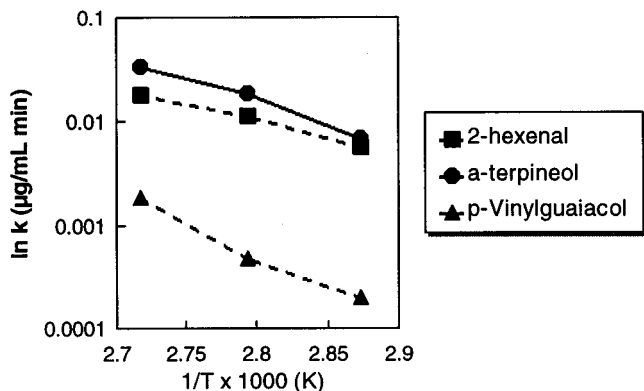
Figure 5. Arrhenius plots for 2-hexenal, α-terpineol, and p-vinylguaiacol.
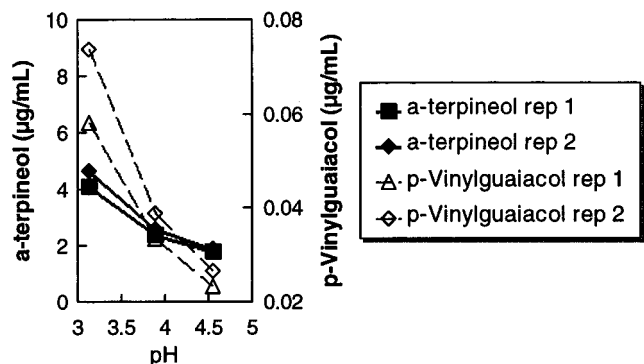
Figure 6. pH effect on α-terpineol and p-vinylguaiacol at T = 95 °C for 15 min.

Table 3. Activation Energy (E_a) and Q_{10} Values for 2-Hexenal, α-Terpineol, and p-Vinylguaiacol Production in the Temperature Range of 75−95 °C

	activation energy		Q_{10} values	
reaction	E_a (cal/mol)	R^2	75−85 °C	85−95 °C
2-hexenal	14831	0.9942	2.0	1.6
α-terpineol	20257	0.9814	2.7	1.8
p-vinylguaiacol	28258	0.9808	2.4	3.9

decreased by ≈0.5 unit as the temperature was increased in 10 °C increments for 2-hexenal. Q_{10} decreased by 0.9 unit for α-terpineol. Contrary to what was expected, a 1.5 unit increase was observed for p-vinylguaiacol as the temperature was increased.

Effect of pH on 2-Hexenal, α-Terpineol, and p-Vinylguaiacol Production. The effect of pH on the generation of α-terpineol and p-vinylguaiacol production with orange juice thermally treated at 95 °C for 15 min is illustrated in Figure 6. pH had the most significant effect on the increase of these two compounds since their concentration doubled at pH 3.1 when compared with values at pH 3.8 and 4.5. This observation is in good agreement with those of Tatum et al. (1975) and Lee and Nagy (1990), who reported that high acidity and high temperatures provide ideal conditions for the release of ferulic acid from its bound forms such as

Table 4. Influence of Oxygen on *d*-Limonene and α-Terpineol (95 °C/60 min)

	d-limonene added (μg/mL)	residual *d*-limonene (μg/mL)	α-terpineol (μg/mL)
no oxygen	0.00	ND	1.11
	84.34	32.24	1.54
	210.34	52.90	2.58
	420.34	258.20	4.25
oxygen	0.00	0.00	1.09
	84.34	51.16	3.99
	210.34	149.91	8.40
	420.34	294.96	11.20

glycosides, esters, and amides, which are the main substrates for p-vinylguaiacol in orange juice, and with those of Blair et al. (1952), who stated that *d*-limonene and linalool degrade via an acid-catalyzed dehydration and hydration pathway (ACHD), yielding α-terpineol in acidic environments. Hexanal, linalool, and valencene remained unaffected by pH (results not shown).

Effect of Oxygen on *d*-Limonene Depletion and α-Terpineol Production. Table 4 represents the relationship of α-terpineol formed as a function of *d*-limonene disappearance in dearated and oxygenated orange juice which was stripped of its volatiles, then fortified with different levels of *d*-limonene, and finally heat treated for 60 min at 95 °C prior to analysis. Oxygen had the most significant effect on the generation of α-terpineol, which is more pronounced with increasing levels of *d*-limonene added to the juice. The quantity of residual *d*-limonene in orange juice was higher in an oxygenated environment than in an anaerobic environment, indicating that the disappearance of *d*-limonene is more pronounced in an anaerobic environment. These observations suggest that the profile of the degradation compounds should be measured directly to estimate an overall change in the quality instead of measuring *d*-limonene loss alone. This observation also shows that dissolved oxygen is the main source of oxygen during the formation α-terpineol from *d*-limonene contrary to what was reported by Shaw et al. (1995), who indicated that the main source of oxygen was derived from water in the juice rather than dissolved oxygen. Furthermore, the increase of α-terpineol could not be correlated with the loss of *d*-limonene as the increase of α-terpineol accounts for ≈10% of *d*-limonene consumed in an oxygenated environment and for <2% in a dearated juice under the experimental conditions observed. This may suggest that *d*-limonene may degrade via other pathways in both the aerobic and anaerobic environments, for instance, autopolymerization to give resinous constituents or other unidentified substances.

Conclusion. Orange juice and many other food products can be subjected to temperature and storage abuse. From a practical standpoint an overall quality change can be expected during distribution and storage of orange juice. Time/temperature abuse experiments revealed that the primary degradation components of orange juice are pseudo-zero-order. The effect of pH was

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significant only for the production of α-terpineol and *p*-vinylguaiacol. The quantity of these compounds was higher at lower pH values, verifying that the reaction is acid-catalyzed (for *p*-vinylguaiacol it is the release of ferulic acid from bound forms that is acid-catalyzed). α-Terpineol levels were strongly correlated to *d*-limonene, and not linalool, whether in an oxygenated or oxygen-reduced environment, indicating that the precursor of α-terpineol is *d*-limonene. The extent of production of α-terpineol was higher under an oxygenated environment; however, *d*-limonene disappeared more quickly in an oxygen-free environment, suggesting that other compounds are produced from *d*-limonene. This study has shown that the most effective method to slow the production of α-terpineol, a malodorous compound identified in orange juice, is to reduce dissolved oxygen.

LITERATURE CITED

Askar, V. A.; Bielig, H. J.; Treptow, H. Aroma changes in orange juice II. Aroma change during the preparation an storage of orange juice in bottles. *Dtsch. Lebensm.-Rundsch.* **1973a**, *69*, 162−167.

Askar, V. A.; Bielig, H. J.; Treptow, H. Aroma changes in orange juice. III. Model experiments on the decrease in linalool and limonene during storage of orange juice in-bottles and cans. *Dtsch. Lebensm.-Rundsch.* **1973b**, *69*, 360−367.

Blair, J. S.; Godar, E. M.; Masters, J. E.; Riester, D. W. Exploratory experiments to identify chemical reactions causing flavor determination during storage of canned orange juice. II. Incompatibility of peel-oil constituents with the acid juice. *Food Res.* **1952**, *17*, 235−260.

Cohen, E.; Berner, R.; Schor, M. Shelf life testing of citrus juices. In *Proceedings of the 19th Symposium of the International Federation of Fruit Juice Producers*, The Hague; IFFJP; Juris Druck and Verlag: Zurich, 1987; pp 401−408.

Kaanane, A.; Kane, D.; Labuza, T. P. Time Temperature Effect on Stability of Moroccan Processed Orange Juice during Storage. *J. Food Sci.* **1988**, *53* (5), 1470−1473, 1489.

Lee, H. S.; Nagy, S. Formation of 4-vinyl guaiol in adversely stored orange juice as measured by an improved HPLC method. *J. Food Sci.* **1990**, *53* (1), 162−164.

Lee, H. S.; Nagy, S. Chemical degradative indicators to monitor the quality of processed and stored citrus products. In *Chemical Markers for Processed and Stored Foods*; Lee, T.-C., Kim, H.-J., Eds; ACS Symposium Series 631; American Chemical Society: Washington, DC, 1996; Chapter 9, pp 86−117.

Lund, D. Heat Processing. In *Principles of Food Science. Part II: Physical Principles of Food Preservation*; Fennema, O. R., Ed.; Dekker: New York, 1975; Chapter 3, pp 31−92.

Lund, E. D.; Bryan, W. L. Commercial orange essance: Comparison of composition and methods of analysis. *J. Food Sci.* **1977**, *42* (2), 385−388.

Marcy, J. E.; Rouseff, R. High-performance liquid chromatographic determination of furfural in orange juice. *J. Agric. Food Chem.* **1984**, *32*, 979−981.

Moshonas, M. G.; Shaw, P. E. Changes in Composition of Volatile Components in Aseptically Packaged Orange Juice during Storage. *J. Agric. Food Chem.* **1989**, *37*, 157−161.

Nagy, S.; Dinsmore, H. L. Relationship of Furfural to Temperature Abuse and Flavor Changes in Commercially Canned Single-Strength Orange Juice. *J. Food Sci.* **1974**, *39*, 1116−1119.

Nagy, S.; Randall, V.; Dinsmore, H. L. Furfural measurements as an index of temperature abuse in stored grapefruit juice. In *Proc. Fla. Hortic. Soc.* **1972**, *85*, 222−225.

Nagy, S.; Rouseff, R. L.; Lee, H. S. Thermally degraded flavors in citrus juice products. In *Thermal Generation of Aromas*; Parliment, T. H., Ho, C.-T., McGorrin, Eds.; ACS Symposium Series 409; American Chemical Society: Washington, DC, 1989; Chapter 31, pp 331−345.

Naim, M.; Striem, B. J.; Kanner, J.; Peleg, H. Potential of ferulic acid as a precursor to -off flavors in stored orange juice. *J. Food Sci.* **1988**, *53* (2), 500−504.

Peleg, H.; Naim, M.; Zehavi, V.; Rousseff, R. L.; Nagy, S. Pathways of 4-vinyl guaicol formation from ferulic acid in model solutions in orange juice. *J. Agric. Food Chem.* **1992**, *40*, 764−767.

Rassis, D.; Saguy, I. S. Kinetics of aseptic concentrated orange juice quality changes during commercial processing and storage. *Int. J. Food Sci. Technol.* **1995**, *30* (2), 191−198.

Shaw, P. E.; Nagy, S.; Rouseff, R. L. The Shelf Life of Citrus Products. In *Shelf Life Studies of Foods and Beverages. Chemical, Biological, Physical and Nutritional Aspects*; Charalambous, G., Ed.; Elsevier Science Publishers: New York, 1993; pp 755−778.

Shaw, P. E.; Moshanas, M. G.; Buslig, B. S. Multivariate analysis for classification of commercial orange juice products by volatile constituents using headspace gas chromatography. In *Fruit Flavors: Biogenesis, Characterization and Authentication*; Rouseff, R. L., Leahy, M. M., Eds.; ACS Symposium Series 596; American Chemical Society: Washington, DC, 1995; Chapter 4, pp 33−47.

Sutherland, J. B.; Tanner, L. A.; Moore, J. D.; Freeman, J. P.; Deck, J.; Williams, A. J. Conversion of Ferulic Acid to 4-Vinylguaiol by Yeasts Isolated from Frozen Concentrated Orange Juice. *J. Food Prot.* **1995**, *58* (11), 1260−1262.

Tatum, J. H.; Nagy, S.; Berry, R. B. Degradation products formed in canned single-strength orange juice during storage. *J. Food Sci.* **1975**, *40*, 707−709.

Toledo, R. T. Thermal Process Calculations. In *Fundamentals of Food Process Engineering*; AVI Publishing: Westport, CT, 1980; Chapter 8, pp 242−281.

Walsh, M.; Rouseff, R. L.; Naim, M. Determination of Furaneol and p-vinylguaiacol in orange juice Employing Differential UV Wavelength and Fluorescence Detection with a Unified Solid-Phase Extraction. *J. Agric. Food Chem.* **1997**, *45*, 1320−1324.