Chapter 22

Chemical Changes in Citrus Juices During Concentration Processes

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This report discusses practical aspects of the effects of heating, evaporation, freeze concentration and reverse osmosis on certain juice constituents, most notably, the volatile flavor constituents. The commercial processes used to remove water from citrus juices have a requirement for thermal treatment of the feed stream to reduce microbial load and inactivate enzymes. While heating stabilizes juice to chemical changes caused by enzymes and microbes, it results in changes to volatile and non-volatile constituents. Also, oxygen reduction prior to heating and/or concentration is important to minimize changes in labile compounds. Most oxygen is removed by vacuum during juice concentration in the evaporator. However, elevated temperatures in the early stages of the evaporator result in some oxygen loss due to ascorbic acid oxidation and other thermally accelerated oxidation reactions.

Strong consumer demand for processed citrus juices is responsible for the very large quantities of concentrates produced and shipped worldwide. The major producers, Brazil and the United States, manufactured approximately 1.32 million metric tons (265 million gal.) of 65% soluble solids (65°B) orange juice concentrate during the 1987 season (1,2). This large amount represents a considerable commitment to concentration, since the final 65°B product has about a 7-fold increase in soluble solids, compared with the single-strength juice. Most of the concentration is performed by high temperature, short time vacuum-steam evaporation; however, recent design improvements in freeze concentration and reverse osmosis have helped to commercialize these technologies in the citrus industry. The following discussion will describe some of the primary juice quality changes resulting from these concentration processes.

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Thermal Treatment

In order to preserve cloudiness and reduce the number of microorganisms which could cause spoilage, citrus juices are heated to temperatures above 90°C for varying lengths of time. The effect of this heating on certain juice constituents has been studied. Ting (3) reviewed thermal effects on nutrients in citrus juices and reported that the severe treatment of heating orange juice to boiling for 15 min resulted in only a 4% loss of ascorbic acid. Other authors also cited the thermal stability of this compound during processing of citrus juices (4).

Perhaps the most easily measured chemical change in citrus juice resulting from thermal treatment is loss of activity of pectinesterases, enzymes responsible for loss of juice turbidity or cloud. Commercial time-temperature conditions vary depending on juice pulp content (5), citrus cultivar, biological maturity, and whether heating is by heat exchanger to stabilize fresh juice or is part of the evaporation process (6). In practice, temperatures vary from 90 to 101°C for times of a few seconds to several minutes (6,7). If such adverse treatment is necessary to inactivate enzymes in juices containing heat-labile constituents, one might expect other chemical changes to occur.

Bitterness. Heating can affect the amount of perceived bitterness in some citrus juices, caused by the presence of the water-insoluble compound, limonin. The insoluble bitter form, limonin, is bound in the pulp and has a more soluble non-bitter hydroxyacid form. Heating of pulp-containing juice is thought to favor chemical equilibrium to the more soluble hydroxyacid, increasing concentration in the juice by extraction from the pulp (8).

Naringin is a bitter flavone glycoside present in grapefruit. Like limonin, it can cause the juice to be very bitter. Naringin is present in the membranes and tissues, is sparingly soluble in cold, but quite soluble in hot water (9). During heat processing, it is extracted into the juice from the insoluble solids and pulp, leading to increased bitterness.

Evaporation

Vacuum evaporation to preserve vitamin C in citrus juice was the subject of a number of early studies (6). Generally, low temperature evaporation of non-heated fresh juice was performed to preserve volatile flavor constituents in the finished product. This technique has now been replaced in the citrus industry by high temperature short time commercial processes, including recovery and concentration of aroma volatiles, which otherwise would be lost during juice concentration (6,7). The most common type of citrus juice evaporator, manufactured by Gulf Machinery, Inc., Clearwater, FL, is referred to as a TASTE (Thermally Accelerated Short Time Evaporator) evaporator. The present process requires juice to be preheated before the first effect of a TASTE evaporator to temperatures as high as 101°C, and remain above 50°C for over 5 min (7). No doubt, such severe heat treatment under vacuum is responsible for quality and flavor changes comparing the fresh juice with the finished 65°B concentrate.

Aroma Changes. Varsel (6) described how the citrus industry practices recovery and use of volatile aromas as essence. However, due to loss of some volatiles during recovery and concentration of the essence, the final product essence does not contain all of the flavor notes of fresh juice. Citrus juice routinely is concentrated from single strength (10°B) to above 65°B. At the highest concentration factor, there is almost no aroma present which could serve to identify the fruit cultivar. In a typical 4-effect commercial citrus juice evaporator, the most volatile aromas and a high percentage of the terpenes have been removed at about 25% evaporation of the initial juice volume (10). Less than this volume results in low recovery of poorly volatile and azeotropic compounds; however, greater than 25% could mean longer exposure of heat sensitive compounds to higher temperatures.

Aroma Quantitation. The terpene fraction of the aroma is mostly S-(+)-limonene and can be used to give an indication of aroma stripping loss or efficiency during concentration. For example, Mannheim et al. (11) reported that at a temperature of 80°C in an evaporator, with 29% of the juice evaporated, the stripped juice contained only 9 ppm limonene of the original 110 ppm in the fresh juice. Here one might caution that measuring the limonene is simple and quantitative (12), but may not accurately represent separation and recovery of more highly volatile or less volatile aromas.

Quantitation and identification of aroma compounds in fruit juices has been a subject of many studies (13). For citrus, single strength juices from extractors have a range of limonene concentration from 0.02 to 0.05% by volume. However, the limonene content in juices reconstituted from evaporator pumpout is usually adjusted to less than 0.02% for best flavor quality. The content of 65°B evaporator pump-out is less than 0.003% limonene. Even though limonene can be reliably quantitated, other juice aroma constituents are probably more important to the flavor. However, quantifying these compounds for the purpose of studying processing changes or chemical changes in products has been at least as complex as identifying them.

Oxygen and Ascorbic Acid. Thermal abuse of juice in an evaporator is more severe than if the juice is pasteurized and cooled in a heat exchanger. Such abuse can result in noticeable chemical browning of the product stream. This browning in citrus juices has been, in part, linked to ascorbic acid loss for stored juices (14). Browning is more obvious for juices from lemon or white grapefruit, which do not benefit from masking pigments. Thermal treatment of grapefruit juice results in a predictable degree of browning, according to one study (15). In another report, the effects of heating during grapefruit juice concentration were related to loss of ascorbic acid and also shown to be predictable (16). An interesting study also suggested that the thermal history or degree of heating of orange juice might be predicted using a bacterial mutagenicity assay specific for some heat-induced browning products (17). However, large variations in some processing operations, or juice properties (e.g. pulp content), may make application of prediction equations difficult.

Oxygen and Ascorbic Acid Loss in TASTE Evaporators

Ascorbic acid degradation has both oxidative and non-oxidative roots. Chen (7) traced the temperature history of juice during concentration in a typical TASTE evaporator. Nagy and Smoot (18) gave extensive data on ascorbic acid loss for orange juice stored at various temperatures in hermetic containers. Calculated ascorbic acid loss based on temperature values of Chen and degradation rates from Nagy and Smoot are given in Table I. Thermally mediated ascorbic acid loss during evaporation would total < 0.02% of the ascorbic acid initially in the juice.

Table I. Anaerobic Destruction of Ascorbic Acid in Orange Juice from the Extractor to Various Stages in a TASTE Evaporator

Stage	Average Temperature °C	Time in Stage Sec	Reaction* Constant (k) %/Sec	Ascorbic** Acid Loss mg/100 ml
Extractor	25	40	2.2×10^{-7}	5.3×10^{-6}
1	46	40	3.0×10^{-6}	7.2×10^{-5}
Preheater	86	10	1.8×10^{-4}	1.1×10^{-3}
2	88	45	2.2×10^{-4}	5.9×10^{-3}
3	76	45	6.8×10^{-5}	1.8×10^{-3}
4	63	65	1.8×10^{-5}	7.1×10^{-4}
5	48	70	3.6×10^{-6}	1.5×10^{-4}
6	42	75	1.9×10^{-6}	8.4×10^{-5}
7	42	80	1.9×10^{-6}	9.0×10^{-5}
				0.01 mg/100 ml

*Based on Arrhenius equation $\ln k = 18.28 - 7709 \cdot 1/T$

**Based on an initial ascorbic acid concentration of 60 mg/100 ml

The oxygen concentration in juice at various stages of a TASTE evaporator is shown in Figure 1. Stage 3 readings identified a vacuum leak. There was no measurable oxygen beyond the 5th stage. Reaction constants for oxidative ascorbic acid loss at various temperatures were determined by Sadler (19). Temperature values of Chen (7) and kinetic solubility values of Sadler (20) were used to calculate oxidation losses in each evaporator stage (Table II). Although oxygen was flashed from the system early in evaporation, oxidative ascorbic acid losses (0.37 mg/100 ml) were over 30 times greater than non-oxidative degradation. This is consistent with previous research which indicated oxidative ascorbic acid losses in citrus ranged from 10 (21) to 1000 (22) times faster than destruction through the anaerobic pathway. Even if all dissolved oxygen was devoted to ascorbic acid degradation, only about 4 mg of ascorbic acid would be lost.

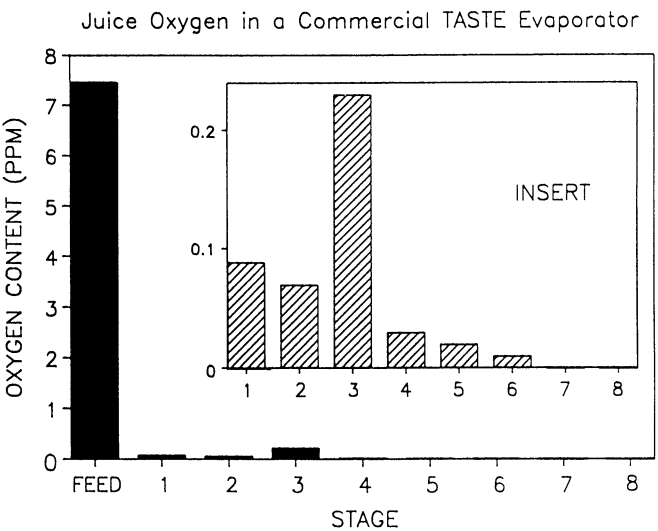

Figure 1. Oxygen content in the feed juice and various stages (1-8) of a TASTE citrus juice evaporator.

Table II. Values for Aerobic Destruction of Ascorbic Acid in Orange Juice from the Extractor to Various Stages in a TASTE Evaporator

Stage	Avg. Temp. °C	Time in Stage (Sec)	Reaction* Constant (k) %/Sec	Ascorbic** Acid Loss mg/100 ml	Oxygen Content Entering Stage ppm
Extractor	25	40	5.1×10^{-4}	0.01	7.47
1	46	40	2.8×10^{-3}	0.07	7.42
Preheater	86	10	4.1×10^{-2}	0.25	7.27
2	88	45	4.6×10^{-2}		6.82
3	76	45	2.1×10^{-2}		0.09
4	63	65	9.2×10^{-3}	0.04	0.07
5	48	70	3.1×10^{-3}		0.03
6	42	75	2.0×10^{-3}		0.02
7	42	80	2.0×10^{-3}		0.01
				0.37 mg/100 ml	

*Based on Arrhenius equation $\ln k = 18.28 - 7709 \cdot 1/T$

**Based on 60 mg/100 ml initial ascorbic acid concentration.

Freeze Concentration

Compared with heat concentration, the superior flavor quality of fruit juices concentrated by freeze concentration has been known for many years (23). Stahl (24) stated for citrus, "juice concentrated by this method possesses a richer fruit flavor than that previously obtained by any other known process, because no volatile flavors or aromas are lost and the chemical changes liable to occur during concentration are reduced to a minimum." More recent reviews of this water removal technique applied to fruit juices concluded the aroma, flavor and nutrient content of products were of superior quality (25,26). The main problem of early freeze concentration designs was loss of soluble sugars during ice removal, resulting in increased cost of concentrated product. This problem has been solved with the commercialization of a multistage process meeting requirements of flavor retention as well as minimal loss of soluble sugars and other juice constituents (27,28). However, high viscosities of the concentrated products at low temperatures limit the degree of concentration and the amount of pulp and insoluble solids which can be present.

To reduce viscosities and favor more efficient water removal at high concentrations, juice pulp reduction by centrifugation is performed prior to freeze concentration of citrus juices. However, this process needs careful control to prevent enzymatic or chemical changes to the juice prior to centrifugation. Also, some important flavor compounds may be bound to or associated with the pulp fraction (29). This might necessitate cautious handling of the separated pulp fraction, particularly if it is to be blended back into a finished product.

Since initial flavor is preserved, the most important consideration for freeze concentration is that feed juices be of very high quality. For example, juice from immature or overmature, or a few rotten fruit, may contain off-flavors which will still be present in the finished product. Also, juice handling practices prior to actual concentration will affect final product quality. For example, holding raw juice for a short time will allow destabilization of the cloud by citrus pectinesterase. This process is not reversible by mechanical homogenization or other treatment.

Orange Juice. In a study of some parameters important to freeze concentration of orange juice, horticultural factors related to the fruit, juice handling and the extent of thermal treatment were more important to product quality than the concentration process (30). It is possible to freeze concentrate fresh, unpasteurized juice, followed by thermal treatment to inactivate enzymes. One could expect that chemical and physical changes to the product would be more apparent after concentration than if heating occurred prior to concentration. Increasing temperature from 80 to 111°C did significantly alter the proportion of reducing sugars and sucrose in freeze concentrate (30). However, only slight changes were measured in common juice quality parameters resulting from thermal processing of freeze concentrate at temperatures of 80, 97 and 111°C (Table III).

Table III. Quality Parameters of Fresh Orange Juice Which was Freeze Concentrated (FCNP), Then Pasteurized for 6 Sec at 80 (FC80), 97 (FC97) and 111°C (FC111)

	FCNP	FC80	FC97	FC111
°Brix	13.3	13.3	13.3	13.3
Acid (%)	0.85	0.85	0.85	0.85
°B/acid	15.7	15.7	15.7	15.7
Oil (%)	0.020	0.018	0.017	0.016
Vitamin C (mg/100 ml)	43	44	43	41
Color (CR)	21.7	21.2	20.8	22.5
(CY)	64.6	64.2	64.6	65.3
(N)	33.2	33.1	33.1	33.4
Pulp (%)	1	1	1	1
Enzyme (PEU)	0.5	0.1	0	0
Viscosity (mPa · s)	3	3	4	4

Data compiled from reference 30.

Orange juice quality parameters may not show obvious change resulting from heat treatment; but sensory changes are more readily detected. Flavor panelists asked to rank the concentrates listed in Table III according to degree of fresh juice or processed flavor were capable of identifying the sample with the most severe heat treatment (111°C), but could not distinguish between concentrates listed in Table IV heated to 80 and 97°C (30).

Table IV. Mean and Standard Deviations for Sensory Ranking Fresh Concentrated Orange Juice Which was Pasteurized for 6 Sec at 80 (FC80), 97 (FC97) and 111°C (FC111)

Ranking	Sample	Evaluations	Mean	Std. Dev.
Most fresh	FC80	15	1.5 c	0.64
	FC97	15	1.7 c	0.70
	FC111	15	2.8 d	0.41
Most processed	FC111	15	1.3 e	0.72
	FC97	15	2.3 f	0.62
	FC80	15	2.3 f	0.71

Means with similar letters are not significantly different. Data compiled from reference 30.

Volatile flavor constituents in freeze concentrated orange juice have been studied and gas liquid chromatographic profiles and data are published. Significant studies by researchers from the Procter and Gamble Co. validate the premise that orange juice concentrated by freeze concentration has exceptional aroma and flavor qualities (31-33). Use of GLC techniques for aroma evaluation of freeze concentrated juices may also be used to indicate something of a juice's processing history. For example, losses of the most volatile juice aromas (alcohol, acetaldehyde) may occur during pasteurization or handling, affecting the proportions between compounds with high and low volatilities (30). On the other hand, a very high concentration of alcohol in aroma fractions condensed from juices could be indicative of yeast growth prior to pasteurization.

Grapefruit Juice. Similar to orange juice, freeze concentration of grapefruit juice results in a product with important aroma and flavor properties preserved. Strobel (34) reported that such a concentrate would contain at least 65% of the total volatile aromas present in the fresh juice. These volatile aromas would also need to be in recommended proportions in order for the reconstituted, blended products to be most like the natural juice. Thermal processes to inactivate enzymes prior to freeze concentration of grapefruit juice may also cause changes in the volatile oils and aroma fractions. Too much peel oil (> 0.023%) in grapefruit juice may not be perceived as acceptable quality; however, steam infusion heating and vacuum cooling/stripping to reduce it to an acceptable amount (< 0.012%), also resulted in a corresponding decrease of other aromas (35). Even though grapefruit has different sensory properties than orange juice, the requirement for highest quality feed juice to produce highest quality freeze concentrate holds true.

Reverse Osmosis

Membrane concentration of liquid foods is common in the food industry. This technology will expand to additional applications with development of more heat stable, chemically inert membranes with high water flux properties. Applications using cellulose acetate reverse osmosis membranes to concentrate citrus juices allowed good retention of important water soluble juice constituents, i.e., sugars, acids, minerals; however, there was poor recovery of some flavor compounds significant for juice quality (36,37). Recent membrane processes for fruit juice concentration have effectively dealt with improving retention of aroma compounds and increasing flux rates (38,39).

Pulp-Serum Separation. As for freeze concentration, high viscosity contributes to limiting the degree of concentration which can be achieved by membrane concentration. Since the insoluble solids present in citrus juices contribute significantly to viscosity, one consideration for process development is to separate the pulp from the clear serum, prior to concentration. Because consumers recognize that citrus juices are cloudy and contain pulpy material, it becomes a requirement to blend the separated pulp back with the product concentrate (31).

Pulp-serum separation followed by pulp addition after concentration is not as simple as it may appear. First, while higher flux and concentration factors of the serum portion may be achieved, the soluble solids and water content of the separated pulp is in equilibrium with the single strength feed juice. Thus, dilution of the concentrate will occur when the pulp is mixed, depending on the amount added. Juice handling time and number of steps will also increase. Under commercial conditions, requirements to handle large volumes may then allow microbial or enzymatic changes to occur in the juice or pulp before and during concentration. Examples of such changes could be cloud destabilization by pectinesterase or production of off-flavors by microbial growth. The latter may be important, since membrane concentration processes do not have the benefit of vacuum aroma stripping as does evaporation.

Another question to be considered is how and when does one remove the pulp? Before enzyme stabilization? Or after? If the pulp is removed first, it is more difficult to heat stabilize the pectinesterase bound in the pulp, requiring high temperatures and long hold times (5), not to mention engineering difficulties of handling and heating pulp. If pulp is removed after heating, removal to yield a serum is more difficult and may involve more handling and longer process times. In either case, flavor and quality properties of the pulp fraction can deteriorate.

Finally, there is evidence that desirable flavor constituents are bound to or associated with the pulp fraction in the juice (29,40). This implies that care must be taken to prevent changes to

Table V. Properties of Feed, Concentrate (Conc) and Permeate (Perm) Streams for Orange, Grapefruit and Lemon Juices Concentrated by Reverse Osmosis

		Orange	Grapefruit	Lemon
Concentration (°B)	feed	11.98	8.68	7.70
	conc	25.26	25.06	22.52
	perm	0	0	0
Hexose (%)	feed	10.68	7.94	1.38
	conc*	10.80	7.15	1.38
	perm	0.03	0.05	0.05
Acid (%)	feed	0.89	0.90	4.63
	conc	1.86	2.66	13.30
	perm	0	0	0
Vitamin C (mg/100 ml)	feed	43	46	36
	conc*	43	45	36
	perm	0	0	0
Volatile oil (%) (as limonene)	feed	0.023	0.007	0.035
	conc*	0.019	0.005	0.024
	perm	0	0	0
Pectin (%)	feed	0.033	0.022	0.05
	conc*	0.020	0.026	0.05
	perm	0	0	0
Pulp (% v/v)	feed	10	7	9
	conc*	8	5	8
Viscosity (cs at 30°C)	feed	1.9	1.5	1.8
	conc	3.9	2.7	6.6

*Values measured after dilution to single strength (feed) °B. Data compiled from reference 42.

these labile compounds during processing and holding for blending to the concentrated serum. Also, the contribution of flavor from the pulp to the blended mixture must be considered, since the flavor spectrum of the concentrated serum-pulp product will be dependent on how much pulp is added.

Juice Properties. Reverse osmosis concentration of orange juice has been reported on at least a small commercial scale (41). For present technology considering preconcentration without the alternative of pulp-serum separation, chemical differences between concentrates and feed streams are slight. Juice and concentrate properties listed in Table V verify that the process is mild. Also, juice aroma and mineral constituents retention by commercial membranes has been shown to be very good for orange, grapefruit and lemon juice concentrated in a pilot plant study (42). The potential for using membranes to recover and concentrate citrus essences, oils, flavor chemicals and other by-products has also been a subject of study (43,44). When one considers the advanced state of membrane technology and pool of available information, the logical conclusion is that large-scale commercial concentration of citrus juices and products will be a reality.

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