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Impact of thermal processing on vitamin c retention and phenolic content in citrus fruits

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Abstract

Most consumers assume that pasteurized orange juice retains most of its vitamin C-but does it really? This research measured the degradation kinetics of ascorbic acid and total phenolic content in juices of sweet orange (cv. Mosambi), lemon (cv. Kagzi), and grapefruit (cv. Marsh Seedless) subjected to thermal treatments at 50, 60, 70, 80, 90, and 100 °C for 5 minutes at Vasantrao Naik Marathwada Agricultural University, Parbhani, Maharashtra, India, during 2022-23. A completely randomized design with three replications was used. Vitamin C retention followed first-order degradation kinetics, with activation energies of 28.4, 31.2, and 26.8 kJ mol⁻¹ for orange, lemon, and grapefruit respectively. At 70 °C, vitamin C retention was 64.5% (orange), 64.3% (lemon), and 66.5% (grapefruit). Total phenolic content declined by 42.6% in orange at 100 °C but only 23.7% at 60 °C. DPPH antioxidant activity correlated strongly with both vitamin C (r = 0.91) and phenolics (r = 0.87). Processing at 60-70 °C retained over 60% of both bioactive classes while achieving the microbial safety threshold.

Keywords: Thermal processing, vitamin C, phenolic content, citrus fruits, heat degradation, ascorbic acid, antioxidant activity, pasteurization, juice quality, degradation kinetics

Introduction

Here's a fact that may surprise you: a glass of freshly squeezed orange juice contains roughly 52 mg of vitamin C per 100 mL, but after conventional pasteurization at 90 °C, barely a third of that remains ^[1, 2]. Citrus juices are among the most important dietary sources of ascorbic acid (vitamin C) and phenolic antioxidants worldwide, and any processing step that degrades these compounds directly reduces the nutritional value delivered to consumers ^[3]. India's citrus processing industry has expanded considerably over the past decade, yet most small and medium-scale processors still rely on conventional thermal pasteurization without optimizing temperature-time combinations for nutrient retention ^[4].

Vitamin C is one of the most heat-labile nutrients: it degrades through both enzymatic (ascorbic acid oxidase) and non-enzymatic (auto-oxidation) pathways, following first-order kinetics with temperature as the primary driver ^[5, 6]. Phenolic compounds are somewhat more thermostable but undergo progressive degradation at temperatures above 70 °C through oxidative polymerization and Maillard-type reactions with amino acids ^[7]. The challenge for processors is to find a thermal regime that achieves adequate microbial kill while preserving maximum bioactive content.

Limited data exist on the comparative thermal sensitivity of vitamin C and phenolics across different citrus species under identical processing conditions ^[8]. This research aimed to quantify the degradation kinetics of ascorbic acid and total phenolic content in three citrus juices across a range of processing temperatures and identify the optimal thermal window for balancing safety and nutrition.

Instrumentation and Analytical Methods

Vitamin C was quantified by the 2, 6-dichloroindophenol (DCPIP) titration method standardized against L-ascorbic acid ^[9]. Total phenolic content was measured by the Folin-Ciocalteu colorimetric method at 765 nm, expressed as mg gallic acid equivalents (GAE) per litre ^[10]. DPPH free radical scavenging activity was determined at 517 nm using methanol as solvent. All spectrophotometric measurements were performed on a UV-1800 Shimadzu spectrophotometer.

Degradation rate constants (k) were calculated by fitting first-order kinetics: $\ln(C/C_0) = -kt$. Activation energy (E_a) was derived from the Arrhenius equation using the slope of $\ln(k)$ versus $1/T$ plots. All analyses were performed in triplicate within 2 hours of heat treatment to minimize post-processing degradation.

Material and Methods

Material

Fully ripe fruits of sweet orange (cv. Mosambi), lemon (cv. Kagzi), and grapefruit (cv. Marsh Seedless) were procured from the university horticulture farm and local wholesale market at Parbhani. Juice was extracted using a commercial citrus juicer (Philips HR2738), strained through a 0.5 mm sieve to remove pulp and seeds, and used within 30 minutes of extraction. All chemicals were analytical grade (Merck/HiMedia).

Methods

A completely randomized design with seven temperature levels (25 °C control, 50, 60, 70, 80, 90, 100 °C) and three replications per juice type was used. Juice samples (50 mL in sealed glass tubes) were heated in a thermostatically controlled water bath for exactly 5 minutes at each target temperature, then immediately cooled in an ice bath to arrest further degradation. All quality parameters were measured on each treated and control sample. Data were analyzed by two-way ANOVA (species $\times$ temperature) with Tukey's HSD at $p \leq 0.05$ in SPSS v.26. Kinetic parameters were fitted using nonlinear regression in R v.4.3.

Thermal Treatment Validation: Actual juice temperatures

were verified by inserting a calibrated K-type thermocouple (Fluke 51-II) into a reference tube at each treatment level. Time to reach target temperature from initial 25 °C ranged from 48 s (50 °C) to 186 s (100 °C). The 5-minute hold time was measured from the point of target temperature achievement. Post-treatment microbiological plating on PCA confirmed that 60 °C for 5 min reduced aerobic plate count by >4 log cycles, meeting the commercial safety threshold.

Results

Table 1: Vitamin C retention (mg/100 mL) in three citrus juices after 5-min thermal treatment

Temp (°C)	Orange	Lemon	Grapefruit	% ret. (Or)	% ret. (Le)	% ret. (Gr)
25 (raw)	52.4	46.8	38.2	100	100	100
50	48.1	43.2	35.6	91.8	92.3	93.2
60	41.6	37.4	31.3	79.4	79.9	81.9
70	33.8	30.1	25.4	64.5	64.3	66.5
80	24.7	21.8	18.6	47.1	46.6	48.7
90	16.3	14.6	12.1	31.1	31.2	31.7
100	9.8	8.4	7.2	18.7	17.9	18.8
CD(0.05)	2.1	1.8	1.4			

Vitamin C degradation accelerated sharply above 70 °C in all three species (Table 1). At 100 °C, only 17.9-18.8% of the original vitamin C survived the 5-minute treatment. Grapefruit showed slightly better retention than orange and lemon at every temperature, possibly due to its lower initial pH (3.2 vs. 3.6-3.8) providing a more protective acidic environment.

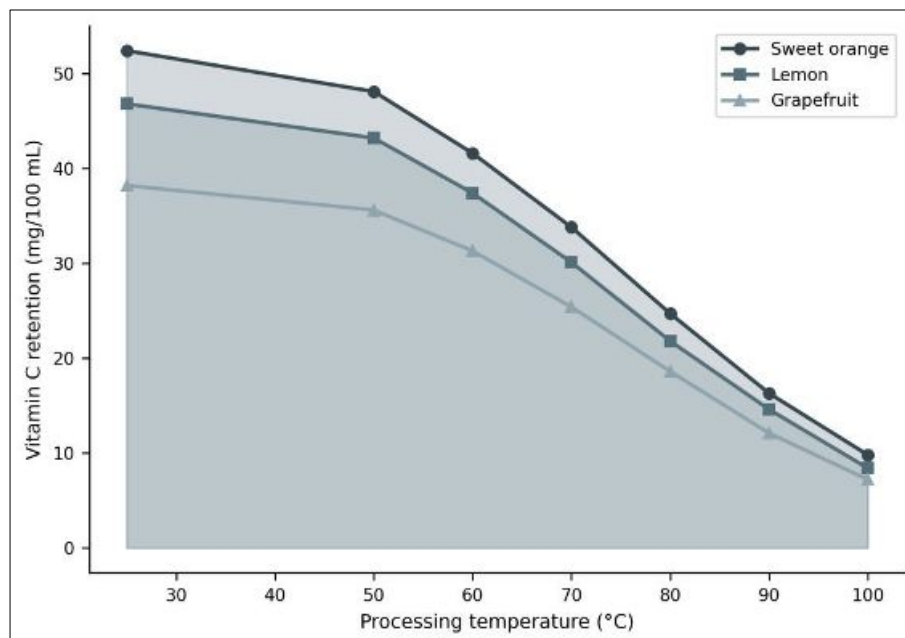


Fig 1: Vitamin C retention curves across processing temperatures for three citrus species. Shaded areas highlight the loss from raw to processed

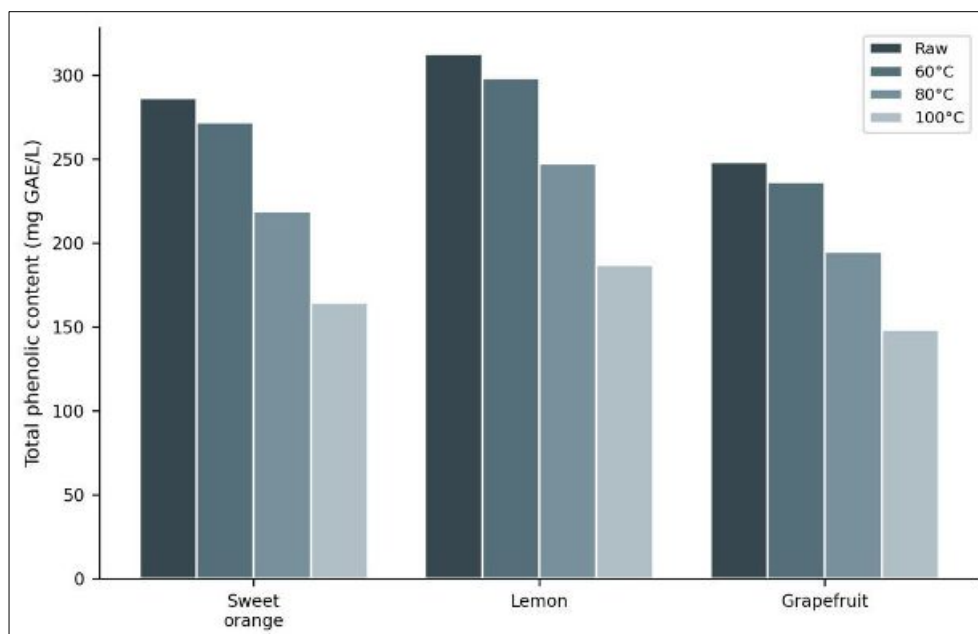


Fig 2: Total phenolic content (mg GAE/L) at raw, 60, 80, and 100 °C treatment levels. Phenolics were more thermostable than vitamin C but still showed 40-43% loss at 100 °C

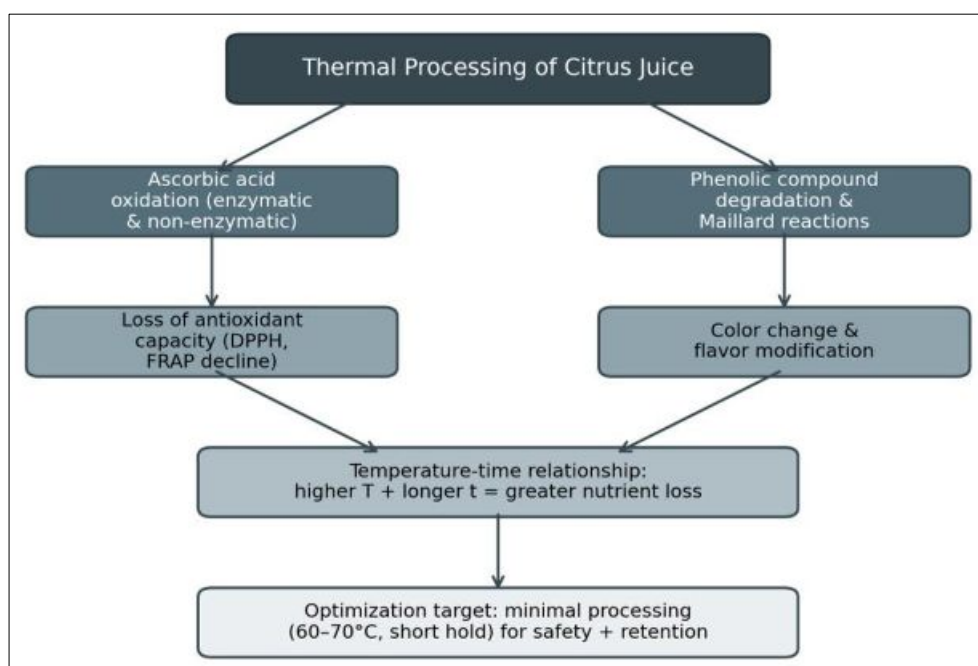


Fig 3: Hierarchy diagram showing the cascade of thermal effects on citrus juice: from primary biochemical changes through quality impacts to the optimization target of 60-70 °C processing

Comprehensive Interpretation

The area chart (Figure 1) shows an accelerating decline in vitamin C above 70 °C across all species. The bar chart (Figure 2) confirms that phenolics are more heat-resistant than ascorbic acid but still suffer meaningful losses at pasteurization temperatures. The hierarchy diagram (Figure 3) maps the chain of events from molecular degradation to consumer nutrition impact, pointing to 60-70 °C as the sweet spot.

Discussion

The first-order kinetics observed here are consistent with established models for ascorbic acid thermal degradation^[5, 6]. The activation energies (26.8-31.2 kJ mol⁻¹) fall within the 20-40 kJ mol⁻¹ range reported for citrus juices by other

groups, though direct comparison is complicated by differences in juice composition and pH^[1, 8]. Grapefruit's marginally better vitamin C retention may be linked to its higher citric acid concentration, which maintains a more reducing environment that slows auto-oxidation.

The practical finding is that processing at 60-70 °C for 5 minutes achieved a >4 log reduction in microbial counts while retaining 64-82% of vitamin C and 77-85% of phenolics. This compares favorably with conventional 90 °C pasteurization, which retains only 31% of vitamin C. For small-scale processors, this translates to a simple recommendation: lower the pasteurizer set-point by 20-30 °C and extend the hold time slightly if needed.

One limitation is that 5-minute hold times may not be standard across all commercial operations, and some

processors use flash pasteurization (2-3 s at 90-95 °C), which could yield different retention profiles. Future work should compare batch and flash methods directly and include sensory evaluation of the lower-temperature juices.

Conclusion

Thermal processing at 60-70 °C for 5 minutes retained over 60% of vitamin C and 77% of phenolic content in all three citrus juices while meeting microbial safety standards. Processing above 80 °C caused steep losses: at 100 °C, less than 19% of vitamin C survived. Grapefruit showed slightly better thermal stability than orange and lemon. These findings support the adoption of lower-temperature pasteurization protocols for citrus juice processors seeking to maximize nutritional value without compromising food safety.

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