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Analysis of Tomato Fruit: Effect of Frozen Storage on Compositional Values—an Inter-laboratory Study

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Tomato culturing trials often result in too many samples to analyse while fresh. The most common technique is to seal them in containers and preserve them by deep-freezing for subsequent analysis. An inter-laboratory study has been made of the effect of freezing for various lengths of time on a number of compositional factors. Tests for soluble solids, dry matter content, electrical conductivity, titratable acidity, potassium, pH, glucose, fructose, sucrose, total N and Vitamin C in frozen tomatoes indicated that the levels of most of these constituents remained relatively constant during frozen storage and were similar to values found in the fruit prior to freezing. When the tomatoes were frozen as a purée it was essential to thaw them in the stabilising/extracting solution used in the Vitamin C analytical procedure, otherwise there was a large loss in ascorbic acid.

1. Introduction

Much has been published about the compositional changes occurring in deep frozen foods¹⁻⁴ during storage in relation to quality retention and nutritive value; similarly, there are extensive data on biochemical changes and compositional values^{5, 6} for fresh tomatoes. However, relatively few data exist on the use of frozen storage for holding material for compositional analysis at a later date; one preliminary report suggests that titratable acidity values for tomatoes may change during frozen storage.⁷

For this reason it was decided to carry out an inter-laboratory study on the possible changes in some compositional values of tomato fruit stored in deep-freeze cabinets over a period of 21 weeks. The participating laboratories were taking part in research projects on tomatoes as part of the Agro-Food Programme of the Standing Committee for Agricultural Research (SCAR) of the EEC.

2. Experimental

The laboratories taking part in the study were located at Station de Technologie C.R.A. d'Avignon, France, at Kinsealy Research Centre, Ireland and at Place Croix du Sud, Louvain-La-Neuve, Belgium and are referred to as Laboratory F, Laboratory Irl and Laboratory B in this paper.

2.1. Design of study

Details of the study were circulated from Laboratory Irl as follows: 2 freezing treatments × 5 testing dates × 5 replicates. The freezing treatments were (a) purée tomato fruit and then cabinet freeze or (b) freeze fruit whole and purée after thawing. Samples of the tomato fruit were tested as purée when fresh (week 0) and from frozen storage on weeks 1, 5, 11 and 19 or as near to these times as convenient.

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2.2. Fruit samples

Tomato fruit of uniform ripeness from the same growing treatment were obtained; a sample comprised 10 fruits giving a total requirement of 500 fruits in each laboratory. Twenty-five lots were packed in groups of 10 (as whole fruit) in triple polythene bags (i.e., 3 bags inside each other) and were frozen in a cabinet freezer at -20 to -25°C . The thickness of the polythene in each bag was $62.5\text{ }\mu\text{m}$. The other 25 lots were puréed with a Kenwood Chef or similar blender for 2–3 min and were packed in the same fashion as the whole fruit. When required for testing, the samples were thawed by placing the bags in hot water (*ca* 80°C). Ten bags of tomato fruit (five of whole fruit and five of purée) were thawed and were tested on each of weeks 1, 5, 11 and 19.

2.3. Tests on the fruit

After thawing, the whole fruits were blended, as described in Section 2.2, to give a purée and tests for percentage soluble solids (%SS), titratable acidity (TA), electrical conductivity (EC), potassium (K) and Vitamin C contents were performed on the purée.

Soluble solids were measured by refractometry. TA was measured using 0.1M NaOH with phenolphthalein as indicator on a 5 g sample of purée diluted with distilled water (expressed as $\text{mEq } 100\text{ g}^{-1}$ purée). Measurements for EC were made at 20°C with a conductivity bridge, and the results expressed as microSiemens; the sample for testing was prepared by diluting 1 part purée with 9 parts distilled water (w/w). The same suspension (filtered) was used for the K estimation (expressed as $\text{mg } 100\text{ g}^{-1}$ purée) which was carried out by flame photometry. Vitamin C content of the purée was determined using the 2,6-dichlorophenolindophenol (DIP) procedure.^{8, 9}

2.4. Additional tests and/or modifications

Tests for the dry matter content and pH of the tomato purée were carried out in Laboratory F, as were two additional tests relating to the Vitamin C estimation. In the first, test samples of puréed fruit, which had been frozen for 11 and 21 weeks, were thawed directly in the extracting medium of the DIP method and the figure obtained for Vitamin C compared with that for puréed tomato fruit thawed prior to the addition of the extracting medium. In the second test, the loss of ascorbic acid during the thawing step was studied using a spectrofluorometric method¹⁰ which allowed the assay of both ascorbic and dehydroascorbic acid. The samples for this test were prepared by crushing tomato fruit under liquid nitrogen. Aliquots (5 g) were thawed and left at 40°C for 0–35 min before extraction.

In Laboratory B, tests were carried out on tomatoes which were frozen whole and then puréed after thawing; no tests were done on frozen purée. The purée made from the whole thawed tomatoes was not tested directly except for the dry matter estimation; the purée was hand pressed in a double layer of cheesecloth to yield juice which was used for all the other estimations. Soluble solids were not measured in Laboratory B but tests were carried out for total N (Kjeldahl procedure, expressed as $\text{mg N } 10\text{ ml}^{-1}$ juice), glucose, fructose and sucrose. The sugars were estimated using the procedure of Blakeney and Mutton.¹¹

3. Results

3.1. Laboratory F

The results (Table 1) show statistically significant changes in all of the factors tested (in the tomato purée) over the frozen storage period with the exception of EC and K content. Titratable acidity values fell over the period while dry matter content increased. Soluble solids content and pH values of the purée were fairly constant throughout with the exception of SS in week 11 when the value was much higher. It should be noted that extreme values were obtained for 5 out of the 7 parameters in Laboratory F in week 11. It seems that this was an effect brought about by a change in the operator on that occasion. The Vitamin C values (Table 1) fluctuated considerably ranging from 8.77 in week 11 to $15.46\text{ mg } 100\text{ g}^{-1}$ in week 21. The low value of 8.77 mg prompted the additional Vitamin C assay outlined above (Section 2.4), where the sample was thawed in the extracting medium of the

Table 1. Tomato fruit composition^a after storage at -20°C (Laboratory F)

Week of test	Soluble solids (%)	Titrateable acidity (mEq 100 g ⁻¹ purée)	pH	Electrical conductivity ^b (μS)	Potassium (mg 100 g ⁻¹ purée)	Vitamin C ^c (mg 100 g ⁻¹ purée)	Dry matter (%)
0	4.7	6.17	4.20	663	0.269	13.61	5.10
1	4.5	6.10	4.26	668	0.261	10.00	5.11
5	4.4	6.10	4.16	671	0.255	10.27	5.16
11	5.2	5.58	4.19	635	0.260	8.77	5.52
21	4.6	5.74	4.19	665	0.248	15.46 ^d	5.46
F-test	$P < 0.01$	$P < 0.01$	$P < 0.01$	$P < 0.05$	NS	$P < 0.01$	$P < 0.01$
s.e. (df=40)	0.13	0.12	0.02	6.0	0.03	1.25	0.9
Frozen as purée	4.6	5.91	4.18	660	0.254	10.28	5.28
Frozen as whole fruit	4.7	5.96	4.22	660	0.262	12.96	5.25
F-test	NS	NS	$P < 0.05$	NS	NS	$P < 0.01$	NS
s.e. (df=40)	0.04	0.02	0.02	0.006	0.04	1.34	0.015
Weeks $\times$ treatment interaction							
F-test	$P < 0.01$	NS	$P < 0.01$	NS	NS	$P < 0.01$	$P < 0.05$
s.e. (df=40)	0.09	0.06	0.01	6.0	0.03	0.87	0.04

^a All tests on puréed fruit.^b One part purée diluted with 9 parts distilled water (w/w).^c Tested by DIP procedure.^d Sample thawed directly in the DIP extracting medium.

DIP method; the value now became 14.16 (mean of week 11 data, Table 2). This procedure was also used in week 21 and is responsible for the high Vitamin C value obtained on that occasion (Table 1).

Statistically significant time $\times$ treatment interactions were found (Laboratory F) in the case of SS, DM, pH and Vitamin C content (Table 1). Inspection of the full data show that the SS values for fruit frozen whole (FFW) were the same as for fruit frozen as a purée (FFP) on the different testing dates with the exception of week 11 when FFW had a higher value. A similar situation was found for pH, the exception being week 21 when FFW had a higher pH than FFP. DM values for FFW were higher than those of FFP in weeks 0, 1 and 21 and lower in weeks 5 and 11. Vitamin C values for FFP were much lower in weeks 1, 5 and 11 than values for FFW. It should be noted that these inter-

Table 2. Effect of thawing procedure on the Vitamin C content (mg 100 g⁻¹) of frozen tomato purée (Laboratory F)

Sample number	Fresh sample	Stored 11 weeks at -20°C		Stored 21 weeks at -20°C
		Thawed slowly	Thawed directly in extracting medium ^a	Thawed directly in extracting medium ^a
1	15.45	4.68	14.64	16.04
2	15.30	4.80	14.28	15.97
3	14.55	3.96	14.40	15.05
4	13.20	5.22	12.72	14.32
5	14.70	5.16	14.76	15.98

^a Used in the 2,6-dichlorophenolindophenol assay.

Figure 1. Change in Vitamin C content of puréed tomato fruit during thawing at 40°C (spectrofluorometric procedure). ○, Ascorbic acid + dehydroascorbic acid; ●, ascorbic acid; △, dehydroascorbic acid.

actions, while statistically significant, were not important in practical terms as they were relatively small effects; the one exception was in the case of Vitamin C content.

The changes in the Vitamin C content of the tomato fruit purée during slow thawing at 40°C are shown in Figure 1. The content of ascorbic acid in the purée fell during the thawing period while that of dehydroascorbic acid increased, indicating that the former was converted to the latter. When both were added together no loss of Vitamin C occurred over the thawing period.

There were only small differences between testing the samples as frozen purée *vs* frozen whole (Table 1) with the exception of Vitamin C content which was lower ($P < 0.01$) in the former.

3.2. Laboratory Irl

The results (Table 3) show that there were statistically significant changes in SS, EC, K, TA and Vitamin C values of the tomato purée over the 19 week frozen storage period. However, inspection

Table 3. Tomato fruit composition^a after storage at -25°C (Laboratory Irl)

Week of test	Soluble solids (%)	Titrateable acidity (mEq 100 g ⁻¹ purée)	Electrical conductivity ^b (μS)	Potassium (mg 100 g ⁻¹ purée)	Vitamin C (mg 100 g ⁻¹ purée)
0	5.0	8.14	720	0.278	8.83
1	5.0	8.44	736	0.264	5.48
5	5.0	8.00	778	0.273	6.98
11	4.9	9.84	729	0.260	6.34
19	5.0	8.04	780	0.270	5.75
F-test	$P < 0.05$	$P < 0.001$	$P < 0.001$	$P < 0.01$	$P < 0.001$
s.e. (df=40)	0.02	0.09	7.6	0.0035	0.23
Frozen as purée	5.0	8.64	750	0.270	2.45
Frozen as whole fruit	5.0	8.36	747	0.268	10.90
F-test	NS	$P < 0.01$	NS	NS	$P < 0.01$
s.e. (df=40)	0.01	0.06	4.83	0.0023	0.14
Weeks × treatment interaction					
F-test	NS	NS	NS	NS	$P < 0.001$
s.e. (df=40)	0.03	0.06	10.8	0.0050	0.32

^a All tests on puréed fruit.

^b One part purée diluted with 9 parts distilled water (w/w).

Table 4. Vitamin C content (mg 100 g⁻¹ purée) of deep frozen whole and puréed tomatoes (Laboratory Irl)

Weeks in storage at -25°C	Tomatoes frozen whole	Tomatoes frozen as purée
0	9.40	8.26
1	10.02	0.94
5	12.94	1.02
11	11.74	0.94
19	10.39	1.11

of the data reveals that, with the exception of Vitamin C, differences were small in practical terms. There were no time $\times$ treatment interactions except in the case of Vitamin C content. This was caused by the very low Vitamin C levels obtained for fruit puréed before freezing compared with that frozen whole (see Table 4). on weeks 1, 5, 11 and 19. Tomato fruit puréed before freezing showed a dramatic fall in Vitamin C content (Table 4) when thawed; presumably this was a function of the thawing procedure rather than the frozen storage treatment as shown in the Laboratory F results in Section 3.1. The data (Table 4) show that no loss of Vitamin C occurred when the tomatoes were frozen whole and puréed after thawing; the thawing was not carried out in the DIP extracting medium in either the whole or purée frozen fruit.

Values for SS, EC and K were the same for fruit frozen whole or as purée (Table 3); however TA values were lower ($P < 0.01$) for fruit frozen whole.

3.3. Laboratory B

The data (Table 5) show no significant changes in the various factors tested in the tomatoes (stored as whole fruit; tests on juice) over the frozen storage period with the exception of fructose content which was higher ($P < 0.01$) at the last testing date and sucrose content which rose in weeks 2 and 6 ($P < 0.05$) and fell again at the 12 and 20 week testing stage.

Table 5. Tomato fruit composition^a after storage at -25°C (Laboratory B)

Week of test	Dry matter content (%)	Titrateable acidity (mEq 100 ml ⁻¹ juice)	Electrical conductivity ^b (μ S)	Total N (mg 10 ml ⁻¹ juice)	Glucose (g 100 ml ⁻¹)	Fructose (g 100 ml ⁻¹)	Sucrose (g 100 ml ⁻¹)	Vitamin C (mg 100 ml ⁻¹ juice)
0	5.1	8.05	707	7.48	1.29	1.21	0.14	8.87
2	5.4	7.59	690	7.42	1.32	1.24	0.24	9.60
6	5.1	7.57	723	7.35	1.35	1.15	0.26	8.95
12	5.3	7.93	690	7.49	1.36	1.22	0.12	9.32
20	5.2	7.82	710	7.59	1.30	1.41	0.12	9.18
F-test	NS	NS	NS	NS	NS	$P < 0.01$	$P < 0.05$	NS
s.e. (df = 20)	0.07	0.23	1.99	0.01	0.0043	0.0138	0.0096	0.041

^a Dry matter content estimated using fruit purée, all other tests on juice pressed from purée.

^b One part juice diluted with 9 parts distilled water (w/w).

4. Discussion

The data from the three laboratories suggest that if tomato fruit are frozen whole there seems to be little change in the various characteristics tested over a period of about 20 weeks with the exception of dry matter content (Laboratory F), sucrose and fructose content (Laboratory B) and titrateable acidity (Laboratories F and Irl). The increase in dry matter content during frozen storage suggests some desiccation. On the other hand it seems unlikely that triple polythene bags would permit this. However, this result suggests that sealed glass containers might have been preferable to polythene. The sucrose levels obtained in Laboratory B were unusually high as values over 0.1% have rarely

been reported. The levels were not checked by an alternative analytical procedure but the relative values from one test date to another are the most relevant in this study.

While potassium was the only element estimated it can be assumed that other elements would behave in the same way and their concentration in the fresh fruit would be similar to that in the frozen.

The findings agree with those of Crivelli *et al.*⁴ for frozen strawberries. They reported values for titratable acidity, pH and total sugars of 0.82%, 3.5 and 4.2%, respectively, in fresh strawberries corresponding values after 5 months storage at -20°C were 0.805%, 3.6 and 3.9% and after 10 months, 0.789%, 3.6 and 4.1%.

Data similar to those found for tomatoes frozen whole were also obtained for tomatoes frozen as purée. It was decided to test fruit puréed before freezing as it is easier to purée fruit at this stage than when whole tomatoes are thawing and releasing a lot of 'drip' liquid. However, this procedure has the disadvantage that it may promote enzyme action. The procedure used for the Vitamin C determination is more important in the case of the frozen purée with emphasis on thawing the purée in the extracting medium of the DIP test procedure. The loss in Vitamin C in frozen puréed tomatoes reported in this study seems to have occurred during thawing and not during the period of frozen storage. Crivelli *et al.*⁴ have shown a loss of Vitamin C in frozen strawberries held at -20°C with values of 38 mg 100 g⁻¹ for the fresh fruit compared with values of 32 mg and 25 mg after 5 and 10 months frozen storage respectively.

Since the method of thawing frozen tomato fruit purée influences the Vitamin C content the practice of using frozen storage as a holding method for tomatoes and other fruit which are to be analysed for their more unstable constituents, e.g. certain vitamins, pigments and other readily oxidisable materials, must be questioned. Therefore, it is desirable that such constituents should be quantified in the fresh fruit where possible. If freezing has to be used as a holding procedure then precautions must be taken to prevent enzymic degradation, exacerbated by maceration of the tissues. Preservation of the material in 80% aqueous ethanol, either in sealed cans or in other containers, may also be used as an alternative to freezing.

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