Food Chemistry **21** (1986) 183–193

Flavour Changes in Gamma Irradiated Grapefruit

Alberto J. Núñez Sellés

National Centre for Scientific Research (CENIC), Apdo. 6990, Havana, Cuba

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Henk Maarse* & Jo M. H. Bemelmans†

TNO Division for Nutrition and Food Research, PO Box 360, 3700 AJ Zeist, The Netherlands

(Received: 13 January, 1986)

ABSTRACT

The effect of gamma irradiation on the volatile components, physico-chemical properties and organoleptic attributes of grapefruit juice extracted from fruits in the dose range 0·50–1·0 kGy was studied. Sensory evaluation of the juice of gamma-irradiated grapefruits after 4 weeks' storage showed that the effect of the irradiation was not greater than that of the storage. The physico-chemical properties (pH and Brix/acidity ratio) of the juice indicate that the radiation effects counteract the storage effect. No radiolytic volatile compounds were found in the extracts of the juice in concentrations above 1 μg litre^{-1}*. Doses of 0·50 and 0·75 kGy did not result in considerable changes of the concentrations of the volatile components, either immediately after irradiation or after being stored for 4 weeks at 12 ± 1°C.*

INTRODUCTION

Cold storage and fumigation are commonly employed in fruit preservation to avoid pest infestations and to extend the shelf-life of these

* To whom correspondence should be addressed.

† Present address: Stichting Bergschot Centrum voor Onderzoek, Postbus 2176, 4800 CD Breda, The Netherlands.

Food Chemistry 0308-8146/86/$03·50

products. In tropical and sub-tropical regions the demand for cold storage is much higher than in temperate regions because of higher ambient temperatures and relative humidity.

Current fumigation treatments prevent fruit infestation but little effect is obtained with respect to decay. Moreover, the chemical residues resulting from this treatment might act as potential carcinogens or phytotoxins. The interest in the use of low-dose gamma irradiation in fruit de-infestation has recently increased since this method does not leave any residue on the fruit; the shelf-life of the product is extended without significant alterations in the fruit properties if an adequate dosage is used. However, changes in texture, colour and flavour can occur at levels needed for de-infestation and/or shelf-life extension. Therefore, in studying the application of irradiation in fruit preservation, attention should be paid to: (i) use of an adequate radiation dosage and (ii) the physiological, physico-chemical and flavour properties of the fruit as compared with that from conventional procedures.

Success with gamma irradiation of grapefruit has been achieved mainly in fruit de-infestation with low-dose treatments (Burditt *et al.*, 1981). Mould decontamination trials have been carried out with dosages higher than 1 kGy, which caused peel injury (Dennison *et al.*, 1966; Riov *et al.*, 1968; Riov & Goren, 1970; Riov, 1971; Riov *et al.*, 1971). Little attention has yet been paid to the effects of irradiation on the flavour properties of grapefruit or grapefruit products (Moshonas & Shaw, 1982). It is necessary to assess these effects within the dosages accepted for grapefruit de-infestation.

MATERIAL AND METHODS

Fruit samples

Fresh grapefruits (*Citrus paradisi* Macfadyen, cv. Marsh seedless) were obtained directly from refrigerated ships in Rotterdam harbour, The Netherlands. Each fruit box was checked by visual inspection and samples were selected that were not affected by pitting, rot or any other external damage. Fruit boxes were stored 1–3 days before irradiation at 12 ± 1°C. One lot (40–64 fruits) in each experiment was not irradiated and was taken as a control sample. All the fruits were obtained from different batches of the same geographic origin (Isla de la Juventud, Cuba) during the 1983 harvest.

Gamma irradiation and storage

Samples were irradiated at the Pilot-Plant for Food Irradiation, Wageningen, The Netherlands, using a cobalt 60 source (plate type) with an activity of 32 kCi. The dose rate was 31 Gy min^{-1} and the dose ratio (D_{max}:D_{min}) 1·2. A bioluminescent method with glutamine was used for dosimetric measurements (Anon, 1977). The samples were packed in polyethylene bags and put in cardboard boxes (60 × 35 × 35 cm) which were rotated in front of the isotopic source at 60 rpm. The irradiation was carried out at room temperature (20°C) with doses of 0·50, 0·75 and 1·0 kGy, respectively, approximately 30 days after harvesting. The irradiated and the control samples were stored in the same room at 12 ± 1°C and a relative humidity of 50%–60% at the Research Institute ITAL, Wageningen, The Netherlands.

Juice extraction

An equatorial zone of the fruit peel was removed (3–5 cm) and the fruits were cut into halves. Each half was covered with adsorbent paper to avoid contamination of the juice with the peel-oil and the halves were carefully reamed on a rotary juice extractor. Seeds, solids and pulp were eliminated by passing the juice through two nylon-screens (40 and 200 mesh). The juice samples thus obtained were subjected to sensory and physico-chemical evaluations and the volatile components were isolated.

Sensory evaluation

Flavour and odour of fruit juice from both control and irradiated samples were assessed in a triangle test by a panel of twelve experienced persons within 3 h after preparation of the juice. Each irradiated sample was evaluated against the control at times 0 and 4 weeks of storage. Assessments were done in separated cabins with dimmed light. The results were checked for statistical differences.

Physico-chemical evaluation

Titratable acidity (measured as citric acid percentage), pH and Brix value (refractometric method at 20°C), according to methods recommended by the IFJU (IFJU, 1985), were measured within 3 h after

preparation of the juice. Four replicates were done for both non-irradiated and irradiated samples.

Isolation of volatile components

A micro steam distillation/solvent extraction procedure was used as previously described (Godefroot *et al.*, 1981; Núñez *et al.*, 1984). A blank determination with 80 ml of deionized water and 2 ml of the solvent mixture (pentane:diethyl ether, 2:1) was carried out. For quantitative analysis, 0·4 microlitre of ethyl hexadecanoate was added to the juice as internal standard. The isolation of the volatile components started within 30 min after preparation of the juice.

Capillary gas chromatography

The isolation was performed twice for each sample and the concentrates were injected (without splitting) in triplicate into a glass capillary column (25 m × 0·25 mm inside diameter) coated with silicone gum SE-30 (Chrompack, The Netherlands). An Intersmat IGC–16 gas chromatograph was used for all analyses. The total amount of volatiles (TAV) was calculated from the sum of the non-corrected concentrations of all the volatiles in milligrams per litre. The volatiles were identified by capillary gas chromatography/mass spectrometry as described above (Núñez *et al.*, 1985).

RESULTS AND DISCUSSION

Some workers have explored the direct effects of irradiation on fruit juices (Gasco *et al.*, 1967; Gasco *et al.*, 1969; Moshonas & Shaw, 1982) but little is known of irradiation effects on juices extracted from irradiated fruits (Nyambatti & Langerak, 1981; Moshonas & Shaw, 1982). Since a large part of citrus harvests is directed to the juice industry, it is important to study the effect of irradiation on juice extracted from irradiated fruit. So far, juice pasteurized by gamma irradiation has not found as wide acceptance as juice pasteurized by conventional heat-treatment.

Table 1 shows the results of the physico-chemical and sensory evaluations of grapefruit juice extracted from irradiated fruits. Only the 1 kGy-sample was different from the control sample in the Brix or the

TABLE 1

Physico-chemical and Sensory Evaluations of Fresh Grapefruit Juice Extracted from Gamma-irradiated Fruits*

Dose (kGy)	*A. Brix (%)*		*B. Acidity (%)*[3]		*C. A:B ratio*		*pH*		*Sensory evaluation*[4]	
	1	*2*	*1*	*2*	*1*	*2*	*1*	*2*	*1*	*2*
0	$8{\cdot}1 \pm 0{\cdot}1^{a}$	$7{\cdot}6 \pm 0{\cdot}2^{a}$	$1{\cdot}2 \pm 0{\cdot}1^{a}$	$1{\cdot}3 \pm 0{\cdot}1^{a}$	$6{\cdot}8 \pm 0{\cdot}1^{b}$	$5{\cdot}8 \pm 0{\cdot}1^{a}$	$2{\cdot}7^{a}$	$3{\cdot}3^{a}$	—	—
0·50	$7{\cdot}8 \pm 0{\cdot}1^{a}$	$8{\cdot}1 \pm 0{\cdot}1^{a}$	$1{\cdot}3 \pm 0{\cdot}1^{a}$	$1{\cdot}2 \pm 0{\cdot}1^{a}$	$6{\cdot}0 \pm 0{\cdot}2^{a}$	$6{\cdot}8 \pm 0{\cdot}2^{b}$	$3{\cdot}0^{b}$	$3{\cdot}3^{a}$	NS[5]	NS
0·75	$7{\cdot}9 \pm 0{\cdot}1^{a}$	$7{\cdot}5 \pm 0{\cdot}2^{a}$	$1{\cdot}3 \pm 0{\cdot}1^{a}$	$1{\cdot}1 \pm 0{\cdot}1^{a}$	$6{\cdot}1 \pm 0{\cdot}2^{a}$	$6{\cdot}8 \pm 0{\cdot}1^{b}$	$3{\cdot}1^{bc}$	$3{\cdot}4^{a}$	NS	NS
1·0	$8{\cdot}8 \pm 0{\cdot}1^{b}$	—	$1{\cdot}5 \pm 0{\cdot}2^{a}$	—	$5{\cdot}9 \pm 0{\cdot}1^{a}$	—	$3{\cdot}2^{c}$	—	$p < 0{\cdot}05$	NS

* Identical letters represent non-significant differences.

[1] Immediately after irradiation. [2] After 4 weeks' storage. [3] Measured as citric acid. [4] Triangle test (odour and flavour). [5] Non-significant.

TABLE 2

Concentration Changes of the Volatile Components of Grapefruit Juice, Immediately after Irradiation (All values expressed in milligrams per litre)

Component	*Dose (kGy)*			
	0	*0·50*	*0·75*	*1·0*
Hydrocarbons				
α-pinene	0·093	0·051	0·035	0·040
myrcene	0·233	0·187	0·206	0·120
β-phellandrene	0·025	0·019	0·078	0·013
dl-limonene	18·2	9·79	11·7	8·06
cis-β-ocimene	0·175	0·143	0·157	0·081
trans-β-ocimene	0·266	0·262	0·231	0·108
α-cubebene	0·766	0·502	0·530	0·320
β-cubebene	0·081	0·051	0·075	0·095
cis-caryophyllene	0·291	0·223	0·318	0·094
trans-caryophyllene	0·233	0·058	0·075	0·089
β-caryophyllene	32·5	25·6	28·3	8·13
δ-cadinene	3·61	2·04	2·17	1·08
Carbonyls				
furfural	0·343	0·218	0·555	0·142
trans-hex-2-enal	0·073	0·001	0·013	0·011
phenylacetaldehyde	0·021	0·010	0·028	0·012
2,6,6-trimethylcyclohex-2-en-1-one	0·227	0·077	0·174	0·050
umbellulone	0·101	0·097	0·159	0·052
carvone	0·148	0·045	0·113	0·063
nootkatone	0·058	0·034	0·035	0·130
Oxides				
3-methyltetrahydropyran	0·075	0·060	0·046	0·022
1-methoxy-1-propoxymethane	0·037	0·028	0·032	0·015
2,2,6-trimethyl-6-vinyltetrahydropyran	0·080	0·072	0·053	0·050
cis-limonene oxide	0·193	0·091	0·067	0·061
cis-anhydrolinalool oxide	0·267	0·212	0·237	0·133
cis-linalool oxide	10·7	7·80	9·01	4·83
Alcohols				
cis-*p*-mentha-2,8-dien-1-ol	0·587	0·413	0·635	0·202
linalool	3·95	3·41	2·48	2·05
α-terpineol	0·125	0·078	0·145	0·036
1-*p*-menthen-9-ol / 3-*p*-menthen-9-ol	0·222	0·108	0·144	0·054
2,6,6-trimethyl-2-vinyl-4-hydroxytetrahydropyran	0·188	0·065	0·118	0·056
2,6,6-trimethyl-2-vinyl-5-hydroxypyran	0·134	0·028	0·053	0·018
p-cymen-α-ol	0·116	0·028	0·053	0·018
β-terpineol	1·07	0·698	1·34	0·467
cis-carveol	1·20	0·437	0·606	0·273
trans-carveol	0·375	0·172	0·198	0·080
geraniol	0·174	0·103	0·081	0·065
β-caryophyllene alcohol	0·485	0·194	0·001	0·234
elemol	0·374	0·158	0·225	0·158
Other oxygen compounds				
ethyl 2-methylbutyrate	0·012	0·001	0·002	0·008
4-vinyl-2-methoxyphenol	0·253	0·235	0·289	0·009
linalyl acetate	0·156	0·093	0·051	0·070

acidity values, which were both higher in the irradiated sample. These values agreed with the sensory evaluation where significant differences ($p < 0{\cdot}05$) for both odour and flavour were only found for the 1 kGy-sample immediately after irradiation. The descriptions given by the assessors varied between 'sweeter' and 'less bitter' for the irradiated samples as compared with the control, suggesting that the irradiation treatment might lead to an improved acceptability of the juice. Moshonas & Shaw (1982) did not find differences in the fresh juice extracted immediately from grapefruits irradiated with 0·25 and 0·50 kGy, respectively. In our experiments, with ranges extended to 1·0 kGy, no differences were found at dose levels up to 0·75. The sensory evaluation after 4 weeks' storage demonstrated that the effects of irradiation on the juice attributes of the 1 kGy-sample were not larger than the effects of storage. Irradiation caused the physico-chemical properties of the juice to counteract the storage effects. While the Brix/acidity ratio of the control sample decreased with storage time, that of the irradiated samples increased. This results in a Brix/acidity ratio of the 1 kGy-sample, immediately after irradiation, equal to that of the control sample after a storage period of 4 weeks.

A study on the chemistry of irradiation flavour should include identification and quantification of the volatile components of control, as well as of irradiated, samples at concentration levels below 1 mg litre^{-1}. We tried to reduce the detection limit of the whole analytical procedure. Good results in reproducibility and recovery efficiency were obtained in the range 0·01–10 mg litre^{-1} (Núñez & Bemelmans, 1984). All volatile components isolated from grapefruit juice in concentrations above 1 μg litre^{-1} could be detected and more than 50% of them could be quantitated by this procedure. No radiolytic volatile compounds above this level were found in the juice.

The results shown in Table 2 demonstrate that the concentration of many volatile components in fruits, irradiated with doses of 0·50 and 0·75 kGy, did not change considerably. Compared with the control sample, a reduction in the concentration was observed for most of the juice components extracted from fruits irradiated with 1 kGy. The TAV values (Table 3) for the 0·50 and 0·75 kGy-samples after 4 weeks' storage were similar to that of the control sample at the time of irradiation. They were considerably higher, however, than for the control sample that had also been stored for 4 weeks, although these results were non-significant owing to the high variability of the control sample ($\delta = 14{\cdot}3$).

TABLE 3

Total Amount of Volatiles (TAV) and Concentrations of Compound Classes in Grapefruit Juice
(All values expressed as $X \pm$ SD in milligrams per litre)*

Batch/Dose (kGy)	*TAV*	*Hydrocarbons*	%	*Carbonyl compounds + oxides*	%	*Other oxygen compounds*	%
			A. Different Non-irradiated Fruit Batches				
September, 1983	$114 \pm 10{\cdot}5^{a}$	$65{\cdot}9 \pm 14{\cdot}9^{a}$	$57{\cdot}9 \pm 3{\cdot}5^{a}$	$29{\cdot}7 \pm 6{\cdot}1^{a}$	$26{\cdot}2 \pm 2{\cdot}2^{a}$	$14{\cdot}8 \pm 8{\cdot}1^{ab}$	$12{\cdot}3 \pm 5{\cdot}5^{a}$
October, 1983	$158 \pm 2{\cdot}3^{b}$	$89{\cdot}3 \pm 15{\cdot}7^{a}$	$62{\cdot}2 \pm 1{\cdot}3^{a}$	$48{\cdot}5 \pm 9{\cdot}6^{a}$	$26{\cdot}5 \pm 0{\cdot}1^{a}$	$26{\cdot}7 \pm 5{\cdot}3^{b}$	$11{\cdot}9 \pm 3{\cdot}8^{a}$
November, 1983	$97{\cdot}0 \pm 4{\cdot}6^{a}$	$67{\cdot}3 \pm 8{\cdot}4^{a}$	$65{\cdot}8 \pm 8{\cdot}4^{a}$	$20{\cdot}6 \pm 5{\cdot}7^{a}$	$20{\cdot}3 \pm 6{\cdot}4^{a}$	$9{\cdot}8 \pm 0{\cdot}1^{a}$	$9{\cdot}5 \pm 0{\cdot}4^{a}$
Average	$123 \pm 28{\cdot}6$	$74{\cdot}1 \pm 13{\cdot}2$	$62{\cdot}1 \pm 4{\cdot}0$	$32{\cdot}9 \pm 14{\cdot}2$	$24{\cdot}3 \pm 3{\cdot}5$	$17{\cdot}1 \pm 8{\cdot}7$	$11{\cdot}2 \pm 1{\cdot}5$
			B. Non-irradiated and Irradiated Fruits of the Same Batch (November, 1983) Immediately After Irradiation				
0	$97{\cdot}0 \pm 4{\cdot}6^{c}$	$67{\cdot}3 \pm 8{\cdot}4^{c}$	$65{\cdot}8 \pm 8{\cdot}4^{a}$	$20{\cdot}6 \pm 5{\cdot}7^{a}$	$20{\cdot}3 \pm 6{\cdot}4^{a}$	$9{\cdot}8 \pm 0{\cdot}1^{b}$	$9{\cdot}5 \pm 0{\cdot}4^{a}$
0·50	$65{\cdot}2 \pm 13{\cdot}7^{abc}$	$46{\cdot}1 \pm 10{\cdot}3^{abc}$	$65{\cdot}0 \pm 2{\cdot}1^{a}$	$15{\cdot}1 \pm 2{\cdot}9^{a}$	$21{\cdot}4 \pm 0{\cdot}1^{a}$	$6{\cdot}3 \pm 2{\cdot}8^{b}$	$8{\cdot}8 \pm 2{\cdot}3^{a}$
0·75	$74{\cdot}1 \pm 4{\cdot}0^{b}$	$52{\cdot}9 \pm 2{\cdot}2^{bc}$	$64{\cdot}8 \pm 0{\cdot}2^{a}$	$17{\cdot}8 \pm 1{\cdot}6^{a}$	$21{\cdot}8 \pm 0{\cdot}7^{a}$	$6{\cdot}7 \pm 1{\cdot}8^{b}$	$8{\cdot}2 \pm 1{\cdot}8^{a}$
1·0	$35{\cdot}7 \pm 2{\cdot}3^{a}$	$23{\cdot}1 \pm 0{\cdot}8^{a}$	$64{\cdot}3 \pm 0{\cdot}6^{a}$	$9{\cdot}3 \pm 0{\cdot}5^{a}$	$21{\cdot}6 \pm 1{\cdot}6^{a}$	$4{\cdot}0 \pm 0{\cdot}1^{a}$	$8{\cdot}9 \pm 0{\cdot}6^{a}$
			C. Non-irradiated and Irradiated Fruits of the Same Batch (November, 1983) After 4 Weeks' Storage				
0	$78{\cdot}1 \pm 14{\cdot}3^{a}$	$49{\cdot}9 \pm 7{\cdot}4^{a}$	$61{\cdot}9 \pm 6{\cdot}2^{a}$	$18{\cdot}2 \pm 4{\cdot}2^{a}$	$22{\cdot}8 \pm 3{\cdot}5^{a}$	$10{\cdot}6 \pm 0{\cdot}1^{a}$	$12{\cdot}5 \pm 0{\cdot}5^{a}$
0·50	$101 \pm 8{\cdot}7^{a}$	$66{\cdot}8 \pm 6{\cdot}7^{a}$	$65{\cdot}1 \pm 1{\cdot}8^{a}$	$26{\cdot}5 \pm 2{\cdot}1^{a}$	$25{\cdot}8 \pm 0{\cdot}3^{a}$	$13{\cdot}9 \pm 3{\cdot}1^{ab}$	$12{\cdot}5 \pm 1{\cdot}9^{a}$
0·75	$104 \pm 2{\cdot}3^{a}$	$65{\cdot}6 \pm 4{\cdot}3^{a}$	$61{\cdot}4 \pm 1{\cdot}5^{a}$	$23{\cdot}7 \pm 2{\cdot}0^{a}$	$20{\cdot}7 \pm 0{\cdot}5^{a}$	$19{\cdot}0 \pm 2{\cdot}1^{b}$	$16{\cdot}8 \pm 2{\cdot}2^{a}$

* Identical letters represent non-significant differences.

† 1 kGy-samples were not processed.

It should be noted that the fraction percentage of compound classes (hydrocarbons, carbonyls, etc.) was not altered by the irradiation treatment, either immediately after irradiation or after 4 weeks' storage. This means that irradiation and storage effects on the TAV values were approximately the same, while no changes in the percentages of the fractions were observed.

The increase in the TAV values for the irradiated samples (0·50 and 0·75 kGy) again suggested extension of the fruit quality during storage (in terms of the TAV, pH and Brix/acidity ratio) by the effect of the irradiation treatment. Comparative data from different non-irradiated fruit batches are shown in Table 3A in order to demonstrate their variations as compared with those observed between non-irradiated and irradiated batches.

The quantitative variation of four volatile components of grapefruit juice are shown in Fig. 1. These changes were compared in terms of dosage-level and storage time to ascertain to what extent they were

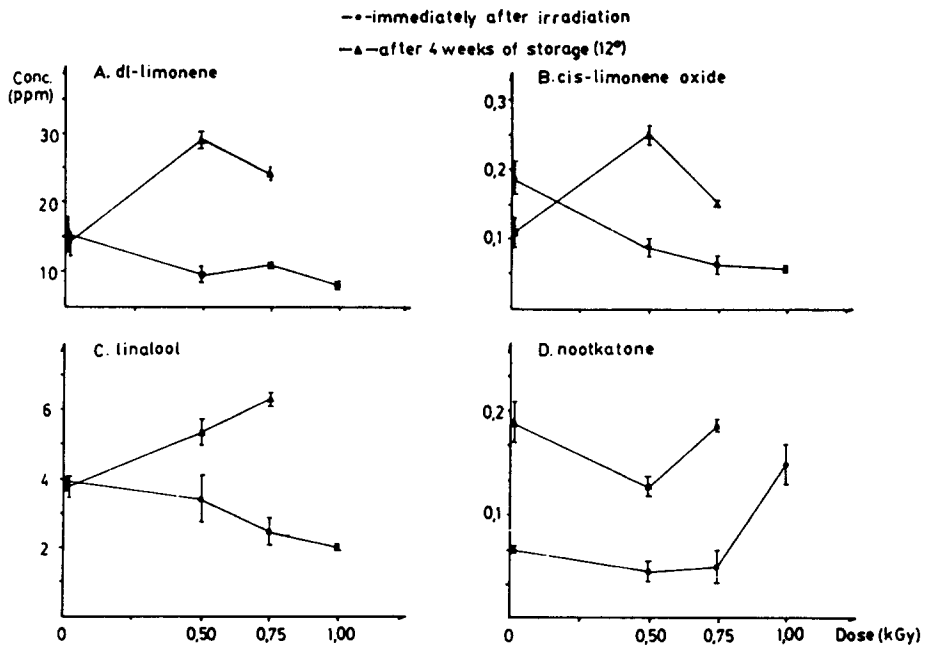

Fig. 1. Quantitative variations of selected volatile components of grapefruit juice in relation to irradiation dose and storage time. A, *dl*-Limonene. B, *cis*-Limonene oxide. C, Linalool. D, Nootkatone.

radiation-induced. The comparisons showed a significant relationship between the dosage-level and the content of linalool after 4 weeks' storage, either with doses of 0·50 or 0·75 kGy. However, other components (*dl*-limonene, *cis*-limonene oxide and nootkatone) showed irregular behaviour upon irradiation. Whether these variations were chemically or biochemically induced needs to be studied further.

ACKNOWLEDGEMENTS

The financial support of the International Atomic Energy Agency (IAEA) granted to one of us (AJN) is gratefully acknowledged. We wish to thank J. Farkas and D. Is. Langerak of the IFFIT Project, Wageningen, The Netherlands, for their collaboration and L. J. van Gemert for his assistance in the sensory evaluation.

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