



# Implications of low-dose e-beam irradiation as a phytosanitary treatment on physicochemical and sensory qualities of grapefruit and lemons during postharvest cold storage

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## ABSTRACT

Since irradiation is a proven decontamination measure, it is essential to monitor the shelf-life and quality of agricultural produce. We evaluated the impacts of low e-beam doses (0, 0.4, 1 kGy) on quality of grapefruit and lemons directly after irradiation as well as during storage (4 °C, 20 d) to simulate transport and market conditions. E-beam irradiation doses of 0.4 kGy and 1 kGy did not alter the weight, texture, total soluble solids (TSS), titratable acidity (TA), sugars, organic acids, vitamin C, narinrutin, hesperidin, and product preference with the exception of a decrease in citric acid of grapefruit and TSS/TA in lemons at 1 kGy. Besides minimal weight loss, firmness did not change in any samples during storage regardless of irradiation doses. Values of TSS and TA remained constant during 20 d of storage in grapefruit and 1 kGy irradiated lemons. Free sugar contents significantly increased, while vitamin C content decreased in both grapefruit and lemons after 10 d. Organic acids and flavonoids underwent delayed (grapefruit) or no changes (lemons) in 1 kGy irradiated stored fruits. Overall, 1 kGy e-beam used for phytosanitation of grapefruit and lemons minimizes quality deterioration during storage.

## 1. Introduction

Foods that are sensitive to heat need technologies that will not expose them to high temperatures typically associated to kill pathogenic microorganisms. As a requirement for global import/export of horticultural commodities, irradiation, being a non-thermal technique is beginning to replace thermal and chemical treatments that are prohibited for sanitization (Hallman, 2012). Hence, there is a rising attention amidst consumers on its safety and health concerns.

Low doses of irradiation for microbial inactivation is a proven intervention technology for commercial applications in the food industry. Fruits are subjected to a minimal dose of ionizing radiations to control quality deteriorations. Notably, irradiation is assumed to extend shelf-life of agricultural produce by 3–5 folds (Arvanitoyannis et al., 2009). Irradiation dose approved for fresh fruits is  $\leq 1$  kGy for shelf-life extension according to the International Atomic Energy Agency (IAEA) and the US Department of Agriculture (USDA) (USDA-APHIS, 2006; IAEA, 2014).

Among highly cultivated agricultural produce, citrus fruits, such as lemons, grapefruit, orange, pummelos, mandarin, and tangerine possess vast economic and health value. In addition, grapefruit and lemons are highly preferred for their unique flavors (Gmitter et al., 2012). Several

researchers have reported the influence of flavonoids on the flavor of citrus fruits (Frydman et al., 2004; Baldwin et al., 2014; Sdiri et al., 2015).

In relation to unknown impact of the irradiation treatment on quality characteristics of grapefruit and lemons, this work aimed to investigate the physicochemical and sensory parameters of e-beam irradiated grapefruit and lemons at phytosanitary doses of 0.4 kGy and 1 kGy as well as during 20 d of storage to simulate transport and market conditions by assessing the loss in weight, strength of fruit peel, total soluble solids, titratable acidity, sugars, organic acids, vitamin C, and flavonoids, along with the sensorial attributes, thus providing information on the export quality of irradiated citrus fruits.

## 2. Materials and methods

### 2.1. Procurement of fruits and irradiation

Grapefruit (*Citrus paradisi*, 374  $\pm$  18 g per fruit, 13  $\pm$  1 fruits per 5 kg box) and lemons (*Citrus limon* (L.) Osbeck, 129  $\pm$  6 g per fruit, 76  $\pm$  2 fruits per 10 kg box) imported from the USA were bought from a market in Daegu, Korea. Citrus fruits were split into 3 groups with 3 boxes each in the same-sized cardboard box (0.36  $\times$  0.26  $\times$  0.12 m)

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and stored for 20 d in a temperature-controlled chamber at 4 °C. One of these groups served as control (non-irradiated).

Two other groups were irradiated with 10 MeV e-beam energy at absorbed doses of 0.4 kGy or 1 kGy using an ELV4 model high-energy linear accelerator with 10 kW power and 100 mA current installed at EBTech, Daejeon, Korea. Alanine dosimeters (0.05 m diameter) attached to the top and bottom of the boxes were used to measure the dosimetry (Bruker Instruments, Rheinstetten, Germany) with  $\pm 5.6\%$  error. The non-irradiated and irradiated citrus samples were stored for 20 d in  $4 \pm 2$  °C.

Analyses were performed per citrus fruit (grapefruit/lemon), irradiation condition (0/0.4/1 kGy), and storage interval of 10 days (0/10/20 d).

## 2.2. Determination of loss in weight and firmness

We measured weight of 10 fruits with a MS105 analytical balance (Mettler Toledo, Greifensee, Switzerland) at 10 d interval up to 20 d and calculated the weight loss % on comparison with initial weight. Ten fruits were selected randomly to evaluate firmness with a Compac-100II rheometer (Sun Scientific, Tokyo, Japan) using adaptor number 5 (0.02 m diameter). The force ( $\text{g}/\text{cm}^2$ ) required to compress 10% of a fruit's diameter was obtained at a strain rate of  $0.01 \text{ m s}^{-1}$ .

## 2.3. Preparation of juice sample

Rinds were removed from ten fruits and the pulp was pressed in a HR-2870 juicer (Philips, Amsterdam, Netherlands). Juice was centrifuged at 4 °C for 20 min at 2500x g, filtered (Whatman No. 4) and kept in  $-18$  °C until analysis.

## 2.4. Determination of soluble solids (TSS), acidity (TA), and TSS/TA ratio

TSS of the juice samples was measured using a Master-M digital refractometer (Atago, Tokyo, Japan). One mL of the sample was added to 9 mL of the distilled water, and the TA was determined by titration with  $0.1 \text{ mol L}^{-1}$  NaOH to a pH of 8.2. TA was calculated as % citric acid.

## 2.5. Determination of free sugars content

Free sugar contents were analyzed using an Agilent 1260 Infinity high-performance liquid chromatography (HPLC; CA, USA) with a Waters carbohydrate column ( $3 \times 0.039 \text{ m}$ ; MA, USA) and an Agilent 1260 refractive index detector. Acetonitrile (80%) was used as mobile phase at a flow rate of  $1 \text{ mL min}^{-1}$ .

## 2.6. Estimation of organic acids content

Organic acids were measured with an Aminex HPX-87H column ( $3 \times 0.075 \text{ m}$ ; Bio-Rad, CA, USA) in an Agilent 1260 Infinity HPLC system and an Agilent 1260 MWD VL UV detector at 210 nm.  $\text{H}_2\text{SO}_4$  ( $5 \text{ mmol L}^{-1}$ ) at a flow rate of  $0.6 \text{ mL min}^{-1}$  was used as mobile phase. Organic acids were identified from retention times of standards.

## 2.7. Determination of vitamin C content

Juice samples were filtered ( $0.45 \mu\text{m}$ ) and injected into the HPLC equipped with a  $\mu\text{Bondapak}$  Waters C18 column ( $3 \times 0.039 \text{ m}$ ) at 30 °C and UV detector at 254 nm.  $\text{H}_2\text{SO}_4$  ( $5 \text{ mmol L}^{-1}$ , pH 2.4) at a flow rate of  $1 \text{ mL min}^{-1}$  was used as the mobile phase. Vitamin C content was expressed as  $\text{g L}^{-1}$ .

## 2.8. Determination of flavonoids content

Fruit peels (5 g) were extracted with methanol (100 mL) following

1 h of sonication. Flavonoids were identified in the HPLC with a ZORBAX Eclipse Plus C18 column ( $1.5 \times 0.046 \text{ m}$ ; Agilent, CA, USA) at 35 °C using a  $0.007 \text{ mL s}^{-1}$  flow rate of the gradient mobile phase (aqueous acetic acid solution:methanol; 90:10 for 20 min, 60:40 at 40 min, 40:60 at 50 min, and 80:20 at 60 min) and UV detection at 285 nm.

## 2.9. Sensory assessment

We performed sensory evaluation to observe the irradiation impact on preference of the product by the end user. Sliced fruits with designated three digit code were given to 20 students. The likings regarding color, smell, taste, and overall acceptability were determined based on 7-point hedonic scale (1 – dislike extremely, 3 – dislike, 4 – fair, 5 – like, 7 – like extremely) in a white light illuminated room. Drinking water was provided to rinse the palate occasionally.

## 2.10. Statistical analysis

All analyses were carried out at least three times. Data were calculated as mean  $\pm$  standard deviation. ANOVA of obtained results was performed in SAS statistical software (v. 8.1, NC, USA) and significance of the mean values at  $p < 0.05$  was calculated using Duncan's multiple range test.

## 3. Results and discussion

### 3.1. Weight loss and firmness of e-beam irradiated and stored grapefruit and lemons

Citrus fruits are perishable commodities and deteriorate quickly after the harvest due to its high respiration rate as well as physiological and mechanical damages (Restuccia et al., 2013). Weight loss in fruits during both irradiation and storage is related to postharvest senescence that modifies its texture (Paul and Pandey, 2014). In both grapefruit and lemons, neither fresh weight nor firmness declined after irradiation with 0.4 kGy or 1 kGy (Table 1). Minimal weight loss was observed throughout storage of all samples. Among them, grapefruit irradiated with 0.4 kGy and lemons irradiated with 1 kGy showed more weight loss as 0.55% and 0.91%, respectively at the end of the storage period. Firmness did not alter during storage in any of the samples.

Shelf-life of 'Shatang' mandarins extended when treated with  $\gamma$ -ray at 0.2–0.4 kGy with least (significant) loss in weight (Zhang et al., 2014) as well as no significant difference in weight loss between irradiated and non-irradiated fruits over 7 d of storage. Jain et al. (2017) observed an instant softening of pulp due to irradiation in 'Chandler' pummelos, while 'Sarawak' pummelos were unaffected by irradiation or storage.

### 3.2. Effect of e-beam irradiation and storage on total soluble solids (TSS), titratable acidity (TA), free sugars, and organic acids of grapefruit and lemons

E-beam irradiation at 0.4 and 1 kGy did not alter TSS and TA of grapefruit (Table 2). Although TSS of lemons were stable at both doses, 1 kGy slightly increased TA. TSS of lemons increased considerably within 10 d of storage except in 1 kGy irradiated samples. During storage TA was affected in both control and 0.4 kGy irradiated lemons, but remained constant in 1 kGy samples. Overall, shelf-life of both grapefruit and lemons were prolonged to 20 d following 1 kGy irradiation, as seen from unaltered TA (%).

Irradiation  $\leq 700 \text{ Gy}$  did not change TSS of an early 'Rio Red' grapefruit (Patil et al., 2004). In contrast, 'Ambersweet' and 'Navel' oranges, as well as 'Sunburst' mandarins subjected to  $\gamma$ -ray at 300, 400, and 450 Gy, respectively showed a decrease in TSS (McDonald et al., 2013). Chisari et al. (2011) observed no changes in the acidity of melon

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