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Food Chemistry

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Combined effects of gamma-irradiation and modified atmosphere packaging on quality of some spices



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ARTICLE INFO

Article history:
Received 23 September 2013
Received in revised form 16 December 2013
Accepted 2 January 2014
Available online 10 January 2014

Keywords:
Gamma-irradiation
Modified atmosphere packaging
Spices
Essential oil
Spice quality

ABSTRACT

Thyme (Thymus vidgaris L.), rosemary (Rosmarinus officinalis L.), black pepper (Piper nigrum L.) and cumin (Cuminum cyminum L.) in ground form were packaged in either air or 100% N₂ and γ -irradiated at 3 different irradiation levels (7 kGy, 12 kGy, 17 kGy). Total viable bacterial count, yeast and mould count, colour, essential oil yield and essential oil composition were determined. Microbial load was not detectable after 12 kGy irradiation of all samples. Irradiation resulted in significant changes in colour values of rosemary and black pepper. The discolouration of the irradiated black pepper was lower in modified atmosphere packaging (MAP) compared to air packaging. Essential oil yield of irradiated black pepper and cumin was lower in air packaging compared to MAP. Gamma-irradiation generally decreased monoterpenes and increased oxygenated compounds, but the effect was lower in MAP. Overall, spices should be irradiated under an O_2 -free atmosphere to minimise quality deterioration.

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1. Introduction

Spices are added to food in order to improve flavour and colour. Thyme, rosemary, black pepper and cumin are some of the most frequently used spices in Turkey. Turkey is one of the main exporter countries of cumin (Weiss, 2002) and a major area for cultivation of thyme and rosemary (Shylaja & Peter, 2004). Spices and herbs are highly susceptible to microbial contamination (McKee, 1995). An appropriate preservation and sterilisation technique should be used in order to maintain shelf life, quality and safety of spices. Gamma-irradiation is one of the commercial methods used in decontamination and sterilisation of spices. Gamma-irradiation of spices and herbs up to 10 kGy (maximum) is approved for decontamination of spices and herbs in the European Union (Directive 1999/3/EC, 1999) and Turkey (Gıda ışınlama yönetmeliği, 1999), whereas the maximum dose is 30 kGy in USA (FDA, 2012) and Australia (Standard 1.5.3, 2012). Food irradiation is widely used, and its applications have been reviewed (Farkas, 1998; Farkas & Mohacsi-Farkas, 2011).

Irradiation is mainly used in spices for sterilisation purposes, but it can have adverse effects on quality of these products through oxidative reactions. For instance, loss of spicy aroma has been reported after irradiation of a meatball preparation containing ground beef and spice mixture (Gunes, Ozturk, Yilmaz, & Ozcelik, 2011; Karadag & Gunes, 2008). Gamma-irradiation increased

oxidation and International Commission on Illumination (CIE) a^* and b^* colour values in several spices (Polovka & Suhaj, 2013). Irradiation at 10 kGy decreased ascorbate and carotenoid content in black pepper and rosemary, respectively (Calucci et al., 2003). The essential oil content of black pepper apparently was not affected by irradiation at up to 30 kGy (Piggott & Othman, 1993), but gamma-irradiation decreased antioxidant and total phenolic contents of some spices, such as thyme (Gumus, Albayrak, Sagdic, & Arici, 2011), rosemary (Perez, Calderon, & Croci, 2007), black pepper (Suhaj, Rácová, Polovka, & Brezová, 2006) and cumin (Kim et al., 2009). Gamma-irradiation at 5–7.5 kGy was recommended for microbial safety of black pepper (Horvathova, Suhaj, & Polovka, 2007).

A combination of irradiation with other preservation techniques (cold storage, heat, packaging, etc.) can decrease the adverse effects of irradiation on the quality of food products (Lacroix, Ramaswamy, & Marcotte, 2003). Modified atmosphere packaging (MAP) simply alters the headspace gas composition of a food product in the package in order to inhibit microbial growth, decrease degradation of quality, and extend shelf life (Gunes & Kirkin, 2012). Irradiation-induced changes, such as softening, respiration, and lipid oxidation in various fresh produce (cabbage, carrot and endive) and meat products (marinated chicken and seasoned ground beef), were significantly decreased by MAP (Ahn et al., 2005; Bekiroglu, Bagbakar, & Gunes, 2007; Gunes et al., 2011; Lacroix & Lafortune, 2004; Niemira, Fan, & Sokorai, 2005). A search of the literature did not find any study on the combined effects of MAP and irradiation on quality of spices.

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The aim of this study was to evaluate the effects of both gamma-irradiation dose and the atmospheric composition during irradiation, on the essential oil content and colour along with total bacterial and yeast/mould count – of thyme, rosemary, black pepper and cumin.

2. Materials and methods

2.1. Materials

Unsterilised samples of thyme (*Thymus vidgaris* L.), rosemary (*Rosmarinus officinalis* L.), black pepper (*Piper nigrum* L.) and cumin (*Cuminum cyminum* L.) (in ground form) were obtained from a local manufacturer (Bagdat Baharatlari Gida San. ve Tic. Ltd. Sti., Ankara, Turkey). Essential oil standard compounds were obtained from Australian Botanical Products (Hallam, VIC, Australia). Plate count agar (PCA), dichloran rose-bengal chloramphenicol (DRBC) agar, peptone and *n*-hexane were supplied by Merck (Darmstadt, Germany).

2.2. Sample preparation and treatments

The spice samples were obtained in ground form and used as provided. No further size reduction or size classification was applied before any treatment or experiment. Packages of 200 g spices were prepared for two different experimental conditions. For the first, spices were packaged in modified atmosphere with 100% N₂ (MAP) using a high-barrier multilayered (polyethylene terephthalate/polyethylene-ethylene vinyl alcohol copolymer-polyethylene) packaging material. The second involved packaging of the spices in air (AP) using a low-barrier low-density polyethylene packaging material. The headspace of the bags of spice was evacuated, then separate samples were flushed with the target gases (100% N₂) and air) before being sealed using a packaging machine (Multivac C200, Multivac Sepp Haggenmüller GmbH & Co. KG, Wolfertschwenden, Germany). The packaged samples were placed in three same size cardboard boxes randomly, and the boxes labelled for different irradiation doses. Amber 3042 dosimeters (Harwell Dosimeters Ltd, Oxfordshire, UK) were placed on each side of the box to measure the applied dose rate. Samples were gamma-irradiated to achieve doses of 5, 10 or 15 kGy at room temperature in a commercial irradiation facility (Gamma-Pak Sterilizasyon San. ve Tic. A.S., Tekirdag, Turkey) with a 60Co source. The average absorbed doses after irradiation were 7, 12 and 17 kGy, and the average dose rate was measured as 2.0 kGy/h. The minimum and maximum applied doses for 5 kGy were 6.5 and 7.4 kGy, for 10 kGy were 11.5 and 13.2 kGy, and for 15 kGy were 16.3 and 17.2 kGy, respectively. The non-irradiated (0 kGy) samples were used as controls. All packaged samples were prepared from the same batch of spices, and the treatments were randomly assigned to the samples. The gas compositions in the headspace of the packages were checked after packaging and prior to analysis using a gas analyser (CheckMate, PBI Dansensor A/S, Ringsted, Denmark). The irradiated spices were kept at room temperature until analysis. All microbial and colour measurements, and essential oil hydrodistillation processes, were started 24 h after irradiation and completed in less than 3 weeks. Each packaging and irradiation treatment was replicated twice. Each replicate was the average of at least two measurements. At least two measurements were used in microbial and essential oil analyses, and colour measurements were repeated at least five times for each replicate.

2.3. Microbial analysis

A 10 g batch of each sample was homogenised in 90 mL peptone water (1% w/v), and serial dilutions were prepared. The total viable

bacterial count (TVC) and total yeast/mould counts were determined by pour plate method, using PCA and DRBC agar, respectively. The PCA plates were incubated at 35 °C for 24–36 h, and the DRBC plates were incubated at 25 °C for 3–5 days. The colonies on agar plates were counted and the result was expressed as log (cfu)/g sample.

2.4. Colour

The values of CIE L^* (lightness), a^* (redness) and b^* (yellowness) were measured using a colour meter (CR-400, Konica Minolta Sensing, Inc., Osaka, Japan). The colour meter was calibrated using a white plate prior to analyses. A sample mass of 20 g was taken into a glass cell (approximately 7 cm diameter), and 5 different measurements from different sample locations were performed and averaged.

2.5. Isolation of essential oils

Spice samples (100 g) mixed with 1600 mL distilled water were subjected to hydrodistillation for 3 h using a Clevenger-type apparatus (Borucam, Istanbul, Turkey) with a 5 mL volumetric scale. The essential oil yield was measured using the volumetric scale of the apparatus. The extracted oil was dried using anhydrous sodium sulfate and stored in amber vials and sealed under nitrogen. A 1000-fold dilution in *n*-hexane was prepared just prior to analysis.

2.6. Gas chromatography-mass spectrometry (GC-MS) analysis

A Varian 3800 GC instrument coupled to a Saturn 2000 mass spectrometer (Varian Inc., Walnut Creek, CA) was used for GC-MS analysis of the essential oils. A Zebron ZB-5 column $(30 \text{ m} \times 0.25 \text{ mm} \text{ I.D.} \times 0.25 \text{ } \mu\text{m} \text{ film thickness, Phenomenex,}$ Torrance, CA) was used for separation. A 1 µL aliquot of the diluted essential oil sample was injected at 10:1 split ratio. Helium was used as carrier gas at 1.2 mL/min. The temperature of the injector was 250 °C. The oven temperature program commenced at 70 °C (hold 6 min), increased at 5 °C/min to 150 °C, then at 10 °C/min to 250 °C, and finally at 20 °C/min to 280 °C (hold 5 min). The mass scan range was m/z 40–400. The peaks were identified using standard compounds when available, and the NIST05 MS library, and quantified using normalised area% based on total ion data. Data were expressed as peak area% of each identified compound to the total sample area, without using any correction factor. In the absence of standard compounds for all compounds, this was appropriate for the profiling comparison here.

2.7. Statistical analysis

The data were subjected to analysis of variance (ANOVA) and Tukey multiple comparison tests using Minitab® 16 Statistical Software (Minitab Inc., State College, PA).

3. Results and discussion

3.1. Microbial analysis

Gamma-irradiation at \geqslant 7 kGy decreased the TVC to undetectable levels (<1 log cfu/g) from the initial levels of 4.5–5.5 log cfu/g in thyme, rosemary and cumin for both packaging treatments (Table 1). Irradiation of black pepper at 7 kGy decreased the TVC to 3.3–3.8 log cfu/g, while 12 kGy irradiation reduced TVC to undetectable levels in both packaging treatments. The initial bacterial load in black pepper was the highest (7 log cfu/g) among the spices

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