Physicochemical-Microbiological Studies on Irradiated Date Fruits with Studying Migration Monomers of Packages Materials

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Abstract

Different food packaging materials (LDPE, HDPE\textsubscript{colorless}, PET and LDPE\textsubscript{blue}) were packaged with dates, and irradiated with γ-rays at 0.0, 1.0, 3.0 and 5.0 kGy. Physical, chemical analysis of Polyethylene low density LDPE-blue layer and colorless--were used. Besides studying the changes of stored dates quality, which extend to nine months under room temperature (22-25°C, 70-75% RH%) and freezing (-3°C), no significant effects were observed in LDPE, as in the permeability of oxygen, carbon dioxide transmission rate and water vapor, or migration tests up to 20.0 kGy; whereas the differences were significant in the mechanical characters. Detection of free radicals using Electron Spin Resonance (ESR) proved presence of free radicals at high dose (20.0 kGy), then disappeared after three weeks. GC-MS analysis of polymers showed produce 18 compounds after irradiation processes, which are volatile or non–volatile compounds at the applied doses. The major constituent was di-n-butylphthalate, which was affected by irradiation. Its concentration was 98.33 % (control), and then decreased to 95.91%, 72.57% by 5.0 and 20.0 kGy, respectively. One of the Radiolytic Products (RPs) are more toxic as bis(2-ethylhexyl)phthalate (0.59%), as mentioned by WHO. Irradiation did not cause significant changes in date’s quality, except the color; only more darkening in color during long storage was observed at room temperature, light color resulted at frozen storage. γ-rays eliminated insects completely and decreased the microbiological contamination in irradiated samples.

Keywords: γ-irradiation; Migration; Fruits; Polyethylene; Dates

Introduction

The packaging materials in contact with the food must be analyzed before using, in order to investigate the potential migrants that could be transferred from the material to the food [1]. A recently approved proposal by the Council of Europe requires the control and analysis of a series of contaminants, such as heavy metals, plasticizers, aromatic amines, polyaromatic hydrocarbons, benzophenone, Diisopropyl-naphthalenes (DiPN’s), Fluorescent Whitening Agents (FWA), Penta-chlorophenol (PCP), and residual solvents, among others [2]. Some of these studies have been carried out, but mostly depend on using food simulants; for instances, solvents at different factors, as temperatures and pH values [3-5]. The interaction for food contact can affect on flavor, aroma, taste of product [6], or the mechanical properties of polymers [7]. The dietary exposures to RPs formed in the materials surveyed were determined to be less than 0.5 μg/kg in the daily diet, less than the threshold of regulation concern level, as per 21 CFR 170.39. However, the author noted that in contrast to the base polymers, the adjutants identified in the survey were not currently listed in 21 CFR 179.45 [8,9].

The conventional method of date conservation after harvest is cold storage at -3°C. This is the most suitable method for the sensitive soft-fruit cultivars, but it is highly energy consuming, as showed by different workers in Israel [9-11]. Recently, more than 40 countries used γ-irradiation on commercial scale for 125 food commodities as spices, fruits, dried vegetables, and other food stuffs.

Therefore, the present work aims to study the effects of γ-rays on used package materials of date fruits, determine the rate of monomer migration by using simulants solvents models, after irradiation of different materials, besides studying the biochemical and quality characters of dates during storage.

Materials and Methods

Irradiation of packaging materials

The used bags practically in food irradiation are multilayer packages, which already contain two craft layers, then polyethylene blue layer, which has contact with food directly. Therefore, LDPE-blue layer was selected for testing migration and GC-MS analysis. In the same time, all over migration test was done for famous package materials LDPE\textsubscript{blue}, HDPE\textsubscript{colorless} and PET. Different types of polymers were irradiated with different doses as followed in table 1.

Irradiation process and storage conditions

Packaged dates were exposed to different doses as 0, 1.0, 3.0 and 5.0 kGy. Dates were γ-irradiated by using Cobalt-60 source (Indian cell), with dose rate10 kGy, hr\textsuperscript{-1} at room temperature (20°C). The irradiation source had been calibrated by the National Physical Laboratory (NPL, Teddington, UK), using the dichromatic dosimetry system. Every dose had two types of bags, multilayer bags (6 bags) and LDPE\textsubscript{colorless} bags (6 bags). Dates were divided to two groups: first one was stored at room temperature (20°C, 70-75% RH%), whereas, the second stored at (-3°C)

| Polyethylene type       | Doses (kGy) | Thickness(mm) |
|-------------------------|-------------|---------------|
| LDPE\textsubscript{colorless} | 0.0, 3.0, 5.0, 10 and 20 | 0.02          |
| LDPE\textsubscript{blue} | 0.0, 3.0, 5.0, 10 and 20 | 0.02          |
| HDPE\textsubscript{colorless} | 0.0, 3.0, 5.0, 10 and 20 | 0.02          |
| PET                     | 0.0, 3.0 and 5.0 | 0.4           |

Table 1: The tested packaging materials.

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Physiochemical and microbiological characters of dates

Date palm (Phoenix dactylifera) fruits, semi dry variety siwi were used in the present experiment. The following characters were studied.

Physical characteristics: The main physical of dates characters as weight loss, Total Soluble Solid (TSS) and texture were measured, according to AOAC [24,25]. Physical observations of insect infestation were examined, recorded and calculated as percentage of the sound dates by staff members of Entomology Lab., NCRRT.

Chemical analysis: Moisture content and pH-values were determined as methods of AOAC [25]. HPLC-analysis of monosaccharide was determined and quantified by Shimadzu Shim-Pack SCR-101N column (7.9 mm×30 cm), using deionized water as the mobile phase (flow rate 1 ml/min at 40°C) and refractive index detection [26]. UV/visible scan were done using the mixtures of dates with UV/visible spectrophotometer Unicam 8625 (Japan) [24].

Microbiological analysis: Total Bacterial Count (TBC) and Total Fungal Count (TFC) were determined using plate count method [27,28].

Statistical analysis: Data were analysed statistically using ANOVA programme, using computer with Duncan analysis [29].

Results

γ-irradiation-effects on packaging materials

Mechanical properties: As shown in table 2, γ-irradiation increased the elongation (E%) of HDPE from 83.83% (control) to 210.50%, after using 5.0 kGy significantly, then E% decreased by increasing doses. Whereas, same values increased with LDPE only, by using 3.0 kGy significantly, but no significant effects were observed with the other doses. In the same time, proportion increase of E% resulted by increasing dose for LDPE-blue at 10.0 kGy, 20.0 kGy, significantly. Same trend was observed for PET at 3.0 kGy, which increased significantly, then decreased at 5.0 kGy. The opposite trend was in parallel with tensile strength values (Mpa).

Effect on permeability: Oxygen, carbon dioxide transmission rate, and water vapor permeability of tested LDPE-blue–common used for dry food irradiation-results as shown in table 3. No significant effect was observed for using γ-rays with different doses on these parameters.

Over all migration test: As shown in table 4, using simulants solutions as iso-octane, acetic acid (3%) for overall migration of irradiated LDPE, proved that no significant effect by irradiation doses up to 20 kGy.

ESR signals for tested samples: The ESR spectra for radical species in irradiated plastic samples are characterized by signal with spectroscopic splitting factor (g-factor) of g∥=2.00663 (Table 5). ESR signal ascribed to free radical induced free radicals of cupper
Effect of irradiation dose on quality of stored dates

A significant effect of irradiation dose on quality of stored dates was observed. Irradiation at 10.0 kGy, 20 kGy, and 30 kGy decreased WT (%) compared with untreated samples, but the brown dates was more darkening. On the contrary, freezing improved color of dates with attractive color for consumers, even at all doses. Also, freezing decreased WT (%) and increased TSS values.

Sugars: Sugar content is considered as the main component in dates, as shown in table 10, HPLC-analysis proved presence of sucrose, glucose, fructose and xylose. Sugars content is present with different concentration (%) and more affected with storage conditions. Sucrose increased rapidly for control samples from 0.64 (at zero time of storage) to 1.24 (room temperature), and 2.75 after storage of nine months at freezing conditions. Generally, freezing caused accumulation sugars, either in irradiated or not irradiated date’s samples. Results showed that storage period and temperature caused significant changes, whereas irradiation not affected most of these characters.

Browning: The recorded values of measuring browning chemically showed that storage temperatures are the main role affecting, as in table 8. Stored samples at room temperature, especially at zero time after nine month at room temperature were more darkening, especially the irradiated samples at higher doses (3.0 and 5.0 kGy). Whereas, freezing dates samples were more attractive in color, regardless of irradiation doses. Only low dose gave the lower value of non-enzymatic browning. These data are in parallel with Hunter color (Table 11), whereas, Lighting (L) values increased from storage at room temperature to more lighting at freezing; for instance, 22.09 to 26.64 (control), 22.54 to 25.64 (1.0 kGy), 23.28 to 28.27 (3.0 kGy), and 22.82 to 25.68 for 5.0 kGy, respectively, at same conditions.

Contamination with insects and microorganisms: Physical examination of dates by using binocular showed that irradiation was more effective on controlling all stages of insects, as in table 12, either during or end of storage. Whereas, freezing keep all dates, but it is more expensive than room temperature. Most of dates in un-irradiated samples contain phases and stages of Ephestia spp insects, as pupa and eggs.

Table 3: Effect of γ-rays doses on the permeability of LDPE-blue.

| Dose (kGy) | O₂ (c/m³) | CO₂ (c/m³) | H₂O(g/m³) |
|------------|-----------|------------|-----------|
| 0.0kGy    | 1090      | 3300       | 3.2       |
| 10kGy     | 1100      | 3400       | 3.14      |
| 20kGy     | 1070      | 3200       | 3.12      |

-Values with the same letter(s) are insignificant difference at 5%

Table 4: Migration test results as changes of weight tested materials.

| Dose (kGy) | Period after irradiation (week) |
|------------|--------------------------------|
|            | 0     | 1     | 2     | 3     |
| 0.0 kGy    | 40.718 | -     | -     | -     |
| 10 kGy     | 61.888 | 40.404 | -     | -     |
| 20 kGy     | 94.257 | 82.34 | 67.172 | 41.15 |

Table 5: ESR-intensity of irradiated plastic during storage.
Discussion

High loss of date quality is recorded annually due to infestation, contamination with microorganisms, either during handling or storage, under unsuitable conditions [9]. Using γ-rays after packaging process for fruits proved the possibility for keeping quality and shelf life in the present work. Different packaging materials are recommended for food irradiation by some authorities, from physical view, not from human health side. Using different recommended package materials (LDPE, HDPE, PET) and LDPE (blue) in food irradiation containing dates after irradiation with 1.0, 3.0, 0.5 kGy, besides untreated samples were done to study the different sides view either for packages or date fruits quality. Physical, chemical analysis of Polyethylene low density LDPE-blue layer and colorless--were used. γ-rays caused some increase

Table 6: GC-MS of migrated compounds from irradiated PET.

| No. | RT  | compound                  | 0.0 kGy | 5.0kGy | 20 kGy |
|-----|-----|---------------------------|---------|--------|--------|
| 1   | 3.47| Octane                    | -       | 12.12  | 1.07   |
| 2   | 9.38| Undecane                  | 0.18    | -      |        |
| 3   | 13.66| Gamma-Himachalene         | 0.28    | -      |        |
| 4   | 13.69| Pentadecane               | -       | 0.69   |        |
| 5   | 14.5| Hexadecane                | 0.78    | 1.56   |        |
| 6   | 14.89| n-pentacosane             | 0.39    | -      |        |
| 7   | 15.3| heptadecane               | 1.48    | 3.62   |        |
| 8   | 15.79| 2-methylheptadecane      | -       | 1.01   |        |
| 9   | 15.93| Octadecane               | 2.82    | 8.13   |        |
| 10  | 16.41| Nanodecane                | -       | 4.79   |        |
| 11  | 16.59| Hexadecanoic acid         | -       | -      | 2.7    |
| 12  | 16.77| Diethylphthalate          | 0.37    | -      |        |
| 13  | 16.88| di-n-butylphthalate       | 98.33%  | 95.91% | 72.57% |
| 14  | 17.44| hexacosane                | -       | 2.25   | 2.77   |
| 15  | 17.97| docosane                  | 0.43    | 2.73   | 2.1    |
| 16  | 19.95| tetracosane               | -       | 0.41   |        |
| 17  | 20.52| pentacosane               | -       | 0.17   |        |
| 18  | 21.01| bis(2-ethylhexyl)phthalate| -       | 0.59   |        |

Table 7: The interaction effect analysis of packaging materials with other factors on quality of stored dates.

| Browning | pH-value | T.S.S. (%) | Moisture (%) | Texture (g/cm²) | Weight loss (%) | Storage period (month) |
|----------|----------|------------|--------------|-----------------|-----------------|------------------------|
| Storage room temperature 1.19 a | 5.746 a | 24.22 a | 8.910 a | 1.870 a | 1.080 a | LDPE(blue) |
| 1.18 a | 5.759 a | 24.02 a | 9.240 a | 1.890 a | 0.530 a | LDPE(colorless) |
| Storage at freezing(-3°C) 1.060 a | 5.980 a | 24.91 a | 9.247 a | 1.295 a | 0.170 a | LDPE(blue) |
| 1.060 a | 5.980 a | 24.91 a | 9.247 a | 1.295 a | 0.170 a | LDPE(blue) |

Table 8: The interaction effect analysis of storage period with other factors on quality of stored dates.

| Browning | pH-values | T.S.S. (%) | Moisture (%) | Texture (g/cm²) | Weight loss (%) | Irradiation dose (kGy) |
|----------|-----------|------------|--------------|-----------------|-----------------|----------------------|
| At room temperature 1.430 b | 5.860 a | 24.30 a | 8.408 a | 1.900 a | 1.510 a | 0.0 |
| 1.440 ab | 5.790 a | 24.40 a | 8.908 a | 1.800 a | 0.560 b | 1.0 |
| 1.590 a | 5.560 a | 23.86 a | 8.930 a | 1.750 a | 0.620 b | 3.0 |
| 1.980 a | 5.816 a | 24.33 a | 9.075 a | 2.030 a | 0.530 b | 5.0 |
| At freezing(-3°C) 0.880 a | 6.010 a | 24.43 c | 9.070 a | 1.250 ab | 0.280 a | 0.0 |
| 0.950 a | 5.950 a | 25.46 a | 9.240 a | 1.430 a | 0.090 b | 1.0 |
| 1.190 a | 5.930 a | 24.66 bc | 9.590 a | 1.210 b | 0.140 b | 3.0 |

Table 9: Interaction analysis of γ- irradiation dose and other factors on quality Siwi dates.
Table 10: Effect of gamma irradiation on sugars content of dates during storage.

| Dose (kGy) | Sucrose% | Glucose% | Fructose% | Xylose% |
|------------|----------|----------|-----------|---------|
| Zero time of storage | | | | |
| 0.0 | 0.64 | 43.4 | 53.3 | 2.63 |
| 1.0 | 1.8 | 44.3 | 52.8 | 1.8 |
| 5.0 | 1.01 | 44.2 | 52.6 | 2.42 |
| After 9 months at room temperature | | | | |
| 0.0 | 1.24 | 44.5 | 53.2 | 1.26 |
| 1.0 | 1.48 | 41.3 | 55.9 | 1.23 |
| 5.0 | 1.43 | 44.8 | 52.6 | 1.19 |
| After 9 months at -3°C | | | | |
| 0.0 | 2.75 | 43.8 | 52.3 | 1.14 |
| 1.0 | 2.53 | 44.2 | 51.5 | 1.01 |
| 5.0 | 2.49 | 42.4 | 51.5 | 0.91 |

Table 11: Hunter parameters (L, a, b) of treated date fruits after 9 months of storage at room temperature and at -18°C.

| Storage | Hunter values |
|---------|---------------|
| RT= Room Temperature | Dose (kGy) 0.0 1.0 3.0 5.0 |
| L       | 22.09 22.54 23.28 22.82 |
| A       | 6.27 5.27 7.21 5.47 |
| B       | 6.27 5.27 7.58 5.55 |
| 3C      | 26.64 25.64 28.27 25.68 |
| a       | 12.79 11.79 13.05 11.83 |
| b       | 14.05 10.05 14.97 10.94 |

Table 12: Percentage of insect infestation for irradiated dates during storage at room temperature.

| Dose (kGy) | Zero time | End of storage |
|------------|-----------|----------------|
| 0          | 0.0 ± 0.0 | 78.4 ± 2.12 |
| 1          | 0.0 ± 0.0 | 92.0 ± 1.00 |
| 3          | 0.0 ± 0.0 | 90.0 ± 0.00 |
| 5          | 0.0 ± 0.0 | 90.0 ± 0.00 |

Table 13: Microorganisms load of irradiated date fruits during storage period.

| Dose (kGy) | Control | 1.0 kGy | 3.0 kGy | 5.0 kGy |
|------------|---------|---------|---------|---------|
| Total bacterial count (T.B.C) | | | | |
| Zero time | 13.89×10⁸ | 3.04×10⁹ | -------- | -------- |
| After 9 months: | | | | |
| Room temperature | 500×10⁸ | 200×10⁸ | 54.5×10⁸ | 32×10⁸ |
| Freezing | 5.5×10⁸ | 3.5×10⁸ | 1×10⁸ | |
| Total fungal count (T.F.C) | | | | |
| Zero time | 52×10 | 16.77×10 | 8.25×10 | |
| After 9 months: | | | | |
| Room temperature | 155×10 | 10×10 | 10.5×10 | 10×10 |
| Freezing | 25×10 | 10×10 | 10×10 | |

Figure 1: Migration without irradiation.
content occupied high content in dates, as shown in table 10, many monosacharides are present as sucrose, glucose, fructose and xylose. These sugars are present with different percentages and more affected upon irradiation are resistant to produce toxic substances for human health. In the same time, using low doses of γ-rays are enough for disinfestations and decontamination at room temperature than freezing from economical side, to keep quality of dates during long storage.

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