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Impact of ionizing radiation on cake from Brazilian macadamia nut (*Macadamia integrifolia*) after oil extraction

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ABSTRACT

Macadamia oil extraction is commonly performed by cold pressing. This process presents low extraction yields, generating partially defatted meal as a byproduct. To take advantage of their important nutritious components, we propose to use the cake after lipid extraction as a food ingredient following the elimination of potentially hazardous microbiological contamination. Food irradiation is a mature, effective, broad spectrum and residue-free technology that can play an important role in food safety and food security. This study presents the impact of gamma irradiation on the residual cake from Brazilian macadamia pressing. An absorbed dose of 5 kGy reduced yeasts, molds and aerobic mesophilic bacteria below admissible maxima. The concentration of important elements like Ca, Se, Mg or Mn appeared higher in the cake than in the nut itself as measured by instrumental neutron activation analysis (INAA).

1. Introduction

Nuts are widely consumed all over the world not only for their taste but also for their health benefits (O'Neil et al., 2010; Tas and Gokmen, 2017). Macadamia nut (*Macadamia integrifolia*) constitutes the edible seed with the highest content of monounsaturated fats. Native from Australia the macadamia nut was introduced in Brazil in 1935, but the first commercial orchard appeared at the end of the seventies. Macadamia nut oil has a high value as it is used in cosmetics as a fragrance fixative and emollient, besides its use as a cooking oil. The extraction process of macadamia oil is commonly performed by cold pressing which presents low extraction yields, generating partially defatted meal as a byproduct.

Macadamia oil cake was proposed for animal feeding (Tiwari and Jha, 2017; Acheampong-Boateng, 2017) as it is practically a waste residue although still rich in nutrients.

Nuts and grains can be contaminated with microorganisms at any stage during production, processing, storage, and distribution (Brar and Danyluk, 2018). Thermal and nonthermal processing for inactivation of microorganisms in nuts can be used, such as heat sterilization, radiation, oxidizing agents and various chemicals (Del Mastro, 2011; Inamura et al., 2012; Van Impe et al., 2018; Macana and Baik, 2018; Saunders et al., 2018; Ravindran and Jaiswal, 2019). This work presents the use of gamma radiation for the decontamination of the Brazilian macadamia residual cake after cold pressing, aiming to be used as a new food ingredient by the food industry.

2. Experimental

2.1. Material

Macadamia nuts and samples of the residue coming from the oil extraction were obtained from Queen Nut Macadamia Company, the main Brazilian producer and distributor. Samples are from different cultivars planted in the same orchard of *Macadamia integrifolia*, varieties from Hawai, Australia and Brazil, mainly cultivars Hawaiian HAES 741, HAES 660, HAES 344, and Brazilian IAC 4–12 B.

2.2. Irradiation

Samples of 100 g were gamma irradiated in polyethylene bags, at average doses of 2.5, 5.0 and 7.5 kGy, dose rate ≤ 1 kGy h^{-1} using a ^{60}Co Gammacell 220, Atomic Energy of Canada Ltd (AECL), dose uniformity factor 1.13 at room temperature. Dosimetry was carried out with cellulose triacetate film dosimeters "CTA-FTR-125" (Fuji Photo Film Co. Ltd).

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2.3. Microbiological analysis

It was carried out on macadamia nut and macadamia cake following the Official Methods of Analysis of AOAC (2000) International, using BioControl and Compact Dry plates for determination and quantification of microorganisms. *Salmonella* sp, coliforms and *Escherichia coli*, molds and yeasts, aerobic mesophiles, and *Bacillus cereus* were evaluated. Analyzes were made according to American Public Health Association (APHA), described in the Compendium of Methods for Microbiological Examination of Food, in the Manual of Methods of Microbiological Analysis of Foods (SILVA, 2001). Tests were performed on macadamia cake about 20 h after irradiation. Total coliforms, thermotolerant coliforms and *Escherichia coli* analyzes followed Most Probable Number (MPN) procedure and the count of molds and yeasts and aerobic mesophiles following Standard Plate Count method.

2.4. Instrumental neutron activation analysis (INAA)

INAA was applied to the multielement characterization (Parry, 2003) of macadamia and macadamia oil cake followed by gamma-ray spectrometry to determine Br, Ca, Cl, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Rb, Se and Zn in samples of around 150 g. As milled macadamia and macadamia cake were homogeneous, no other treatment was done. Five aliquots (approximately 700 mg) of milled macadamia and five aliquots (approximately 300 mg) of macadamia cake were weighed into polyethylene bags. These bags had been cleaned by leaching with a diluted HNO_3 (1:5) solution and purified water (Milli-Q). Certified standard solutions (Spex Certiprep) of Br, Ca, Cl, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Rb, Se and Zn were used to prepare elemental synthetic standards. Aliquots (50–100 µl) were pipetted onto small sheets of analytical filter paper (Whatman N° 42) for neutron irradiation. After drying, these filter papers were placed into polyethylene bags. Two types of neutron irradiations were carried out at the IEA-R1 nuclear research reactor of IPEN-CNEN/SP. First, the sample and standards of the elements Cl, Cu, Mg and Mn were irradiated together in a polyethylene container for 20 s. After a decay time of 2 min the ^{38}Cl, ^{66}Cu and ^{27}Mg were measured in the sample and in the standards. ^{56}Mn was measured after 90 min of decay time. In the second irradiation, the sample and standards (Br, Ca, Co, Cr, Fe, K, Na, Rb, Se and Zn) were irradiated together in an aluminum container for 8 h. The ^{82}Br, ^{47}Ca, ^{42}K and ^{24}Na were measured after 4 to 5 days of decay time, while ^{60}Co, ^{51}Cr, ^{59}Fe, ^{86}Rb, ^{75}Se and ^{65}Zn were measured after, at least 8 days of decay time. The thermal neutron flux utilized was of the order of 1×10^{12} n cm^{-2} s^{-1}. The quality of the standards was previously checked through the analysis of reference materials. The equipment used to measure the gamma radiation was a GX2020 hyperpure Ge detector, coupled to a model 1510 Integrated Signal Processor and MCA 100 System, both from Canberra. The detector used had a resolution (FWHM) of 1.0 keV for 122 keV gamma rays of ^{57}Co and 2.0 keV for 1332 keV gamma-rays of ^{60}Co. Element concentrations were calculated by the comparative method.

2.5. Centesimal composition and fatty acid profile

Homogeneous samples of ground macadamia nut and macadamia cake were analyzed in triplicate. Analyzes of moisture (IAL 12 and 13/IV), ash (IAL 018/IV), and total fat (IAL 032/IV) were undertaken following the Instituto Adolfo Lutz (2008) Analytical Standards. Protein (Method 2001.11) and fiber (Method 991.43) were determined following the Official Methods of Analysis of AOAC International. According to the Technical Regulation on Nutritional Labeling of Packaged Food provided in Resolution RDC No. 360, dated December 23, 2003, from Brazilian Health Regulatory Agency (ANVISA), carbohydrates were calculated as the difference between 100 and sum of the content of proteins, fats, fibers, moisture and ashes. Calorie values were calculated using conversion factors where 1g of protein provides 4 calories, 1g of fat provides 9 calories and 1g of carbohydrate provides 4 calories.

Table 1
Chemical composition (g 100 g^{-1}) of macadamia nut and cake.

Component	Nut	Cake
Protein	8.58 ± 0.15	11.57 ± 0.18
Total fat	50.48 ± 0.51	19.24 ± 0.81
Saturated fatty acids	6.63 ± 0.07	2.49 ± 0.03
Trans fatty acids	ND	ND
Monounsaturated fatty acids	39.15 ± 0.18	14.97 ± 0.03
Polyunsaturated fatty acids	2.48 ± 0.10	0.93 ± 0.00
Humidity	1.54 ± 0.00	2.07 ± 0.01
Ash	1.32 ± 0.01	1.93 ± 0.06
Fibers	0.18	0.93
Carbohydrates	37.90	64.26
Calories (kcal/100g)	640.24	476.48

ND = not detectable. Values are presented as mean ± SD (n = 3) except for fibers. Carbohydrates and calories were calculated.

3. Results and discussion

In a previous paper, the radiation stability of centesimal composition and profile of fatty acids of macadamia cake was established for same dose range (Rao & del Mastro, 2019).

Chemical composition and energetic value of the macadamia nut and cake are displayed in Table 1. The major components were lipids (> 50 g/100g and > 19 g/100g), especially monounsaturated fatty acids (> 39 g/100g and 15 g/100g), carbohydrates, protein, moisture and minerals. According to Mereles et al. (2017), the lipid fractions of nuts are composed mainly of oleic (C18:1) and linoleic (C18:2) fatty acids, with emphasis on the ω-6 to ω-3 relation in macadamia, whose profiles favor the reduction of cardiovascular disease risk (Freitas and Naves, 2010).

A preliminary microbiological evaluation of macadamia nut and cake was performed (Table 2). Pathogens like *Salmonella* sp was absent in both kind of samples, coliforms and *B. cereus* counts were very low and within hygienic satisfactory range.

Samples of macadamia cake were subjected to gamma irradiation and analyzed in triplicate (Fig. 1 and Fig. 2). Yeast and mold count in the control samples and irradiated at 2.5; 5 and 7.5 kGy were, respectively, $(2.7 \pm 0.49) \times 10^5$, $(1.7 \pm 0.10) \times 10^4$, $(2.6 \pm 0.92) \times 10^3$ and < 10. Mesophilic aerobic count in the samples irradiated at the same doses were, respectively, $(8.0 \pm 0.12) \times 10^5$, $(7.9 \pm 0.76) \times 10^4$, $(1.6 \pm 0.14) \times 10^4$ and < 10. Yeast and mold bioburden as well as aerobic mesophilic bacteria were reduced below international admissible limits for trade of nuts (5×10^3 and 5×10^4 respectively), guaranteeing microbiological safety (Brasil, 1997; Leitao, 1988).

The presence of yeasts and molds on dried fruits and nuts can be a public health risk because of the potential for exposure to toxigenic fungi. Mesophilic microorganisms are one of the more general and extensively microbiological indicators of food quality, indicating the adequacy of temperature and sanitation control during processing, transport, and storage.

Some reports have been published on the efficacy of ionizing radiation processing in reducing bioburden in English walnuts (Wilson-Kakashita et al., 1995), cashew almonds (Freire and Del Mastro, 2000), pecan (Taipina et al., 2009) and Brazil nuts (Assunção et al., 2015);

Table 2
Microbiological evaluation of macadamia nut and macadamia cake.

Organism	Nut	cake
Coliforms at 45 °C (UFC/g)	< 10	< 10
Salmonella sp (/25g)	Absent	Absent
Bacillus cereus (UFC/g)	$< 15 \times 10^1$	$< 15 \times 10^1$

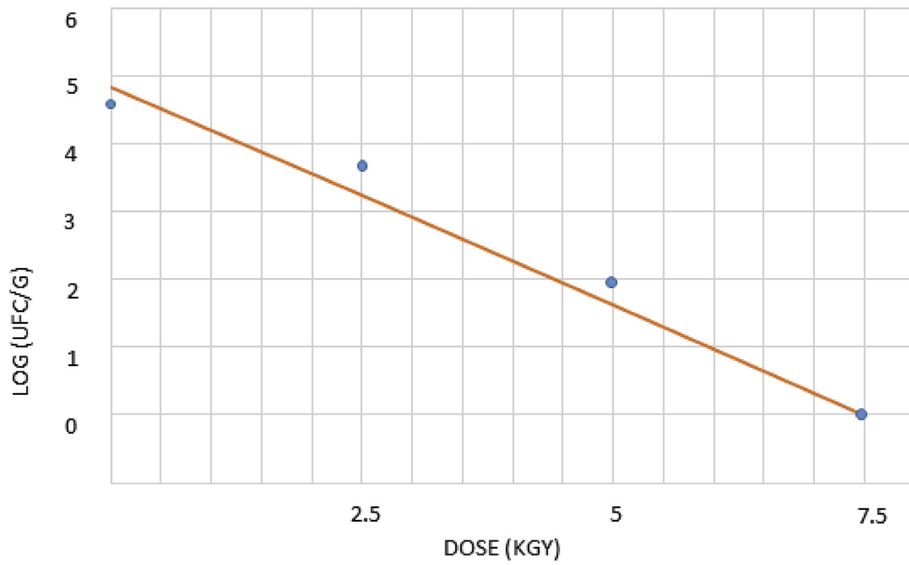

Fig. 1. Molds and yeasts count of macadamia cake as a function of absorbed dose. Linear regression analysis represents the mean best fitting line.

present results on macadamia nuts corroborate data from the literature on the efficiency of radiation on nut decontamination.

Present results are much lower to the Proposed Organisation for Economic Co-operation and Development (OECD) microbiological contamination screening requirements for Total aerobic bacteria (mesophiles) 10^8 CFU/g or mL - indicator of aerobic bacterial contamination - and Yeast and Mold Count that need just being visually monitored (https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_ppp_app-proc_guide_phys-chem-ana_microbial-contaminant-limits.pdf).

INAA was employed to determine elemental concentrations of Br, Ca, Cl, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Rb, Se and Zn in macadamia nut and macadamia cake. Values are presented as means ± SD (n = 3). Our own data and those from the literature were also presented in a comparison displayed in Table 3.

Mereles et al. (2017) also analyzed macro and microminerals of macadamia samples using chemical methods. They also found significant content of Mg, K and Ca which are minerals normally found in high concentrations in nuts. Regarding trace minerals, high concentrations of Fe and Mn were also observed.

The observed values for Ca, Mg, Mn and Cu are in the range

Table 3
Comparison of micronutrient composition (μg. g^{-1}) of macadamia nut and cake data from INAA present results and statistical information from the literature.

Element	nut [a]	nut [b]	nut (NAA)	cake (NAA)
Br	-	-	22.4 ± 0.4	25 ± 3
Ca	668.4	850	447 ± 29	657 ± 135
Cl	-	-	129 ± 21	190 ± 32
Co	-	-	0.10 ± 0.01	0.29 ± 0.04
Cr	-	-	0.35 ± 0.03	ND
Cu	7.2	8	3.0 ± 0.8	4.7 ± 0.1
Fe	23.0	37	26 ± 3	22 ± 4
K	3531.6	3680	4021 ± 453	5201 ± 586
Mg	1265.0	1300	981 ± 126	1549 ± 250
Mn	32.7	41	31.5 ± 1.9	49.7 ± 3.8
Na	21.2	50	5.9 ± 0.2	7.6 ± 0.4
Rb	-	-	13 ± 2	17 ± 3
Se	-	-	42 ± 23	ND
Zn	15.3	13	15.9 ± 0.6	19.9 ± 0.9

ND = not detectable.
[a] Adapted from Mereles et al. (2017).
[b] http://www.nware.com.br/tbca/tbca/.

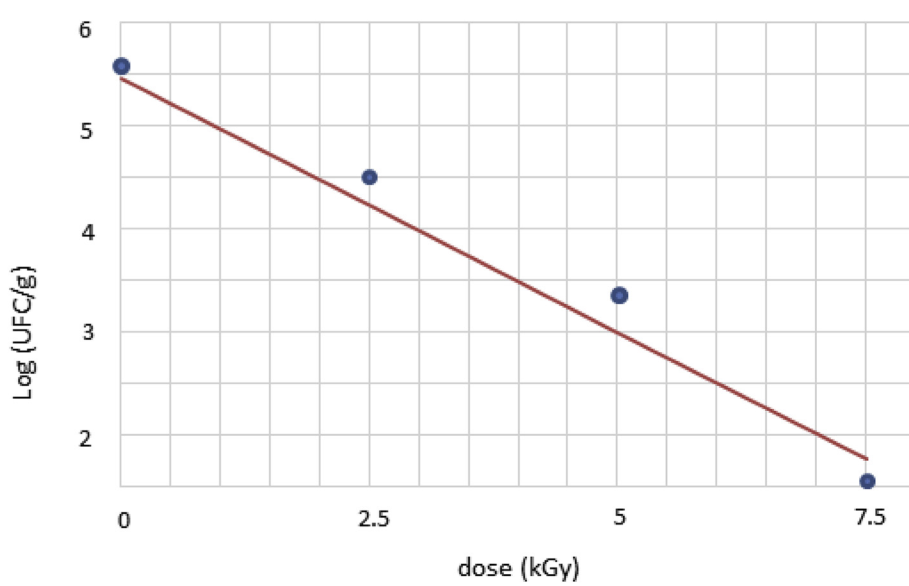

Fig. 2. Aerobic mesophiles counts of macadamia cake as a function of absorbed dose. Linear regression analysis represents the mean best fitting line.

reported by Silva (2003) and the USDA (2016), for raw *M. integrifolia* nuts in different samples of commercial macadamias from Brazil and the United States, respectively. Mineral data obtained with INAA gave a more complete spectrum of elements content of macadamia than other analytical methods. As macadamia cake is very nutritious and still rich in fat it can be used to replace, for instance, egg and dairy ingredients in terms of texture, as moisture and fat content are the two key determining factors for texture creation.

As mineral composition is related to the conditions of each region (minerals are absorbed from the soil), discrepancies among minerals content of nuts like macadamias coming from different region, culture and period of the year are expected.

4. Conclusion

The macadamia cake resulting from the oil extraction showed to be more nutritious than the macadamia nut itself in terms of mineral components such as Ca, K, Mn, Mg and Zn. Since the present samples of Brazilian macadamia cake did not present salmonella or coliform contamination, radiation treatment showed to be a good treatment to reduce the bioburden of molds and yeasts and aerobic mesophiles. From present results, absorbed doses around 5 kGy could be sufficient for safe commercialization and human consumption of the macadamia oil meal. Although more tests must be performed to assure the exact dose value, radiation treatment is able to reduce the bioburden in macadamia cake.

CRediT authorship contribution statement

Ina P. Rao: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing - original draft, Writing - review & editing. **Maria J.A. Armelin:** Methodology, Validation, Formal analysis, Investigation. **Nelida L. del Mastro:** Conceptualization, Investigation, Writing - original draft, Writing - review & editing, Supervision, Project administration.

Declaration of competing interest

I declare that there is no conflict of interest in publishing this article.

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