

Method for Analysis and Study of Migration of Lead, Cadmium, Mercury and Arsenic from Polypropylene Packaging into Ice Cream and Simulant

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Abstract The aim of this study was to propose a method for analysis of lead (Pb), cadmium (Cd), mercury (Hg) and arsenic (As) in polypropylene packaging intended for contact with ice cream and migration of these elements using the food itself and 3 % acetic acid as food simulant. Ten commercial samples of ice cream marketed in Campinas-São Paulo (Brazil) were used for the analysis. Both the packaging and ice cream were digested in high-pressure ashing equipment (HPA), as a new proposal to the sample preparation and the migration of the elements into stimulant directly in the solution. The determinations were carried out using an inductively coupled plasma optical emission spectroscopy (ICP-OES) and two different nebulizers (cross flow and Miramist, when necessary). The results obtained showed that the levels of Hg and As of all packaging were below the limit of quantification (LOQ) corresponding to 1.0 and 1.7 $\mu\text{g L}^{-1}$, respectively. The highest levels of Cd and Pb were 0.45 and 1.64 mg kg^{-1} , respectively. The migration of these elements into the ice cream after 365 days of contact at $-20\text{ }^{\circ}\text{C}$ was below the LOQs for all the samples assessed. The results of specific migration into 3 % acetic acid simulant showed concentrations of Cd, Hg and As below 5, 5 and 10 $\mu\text{g kg}^{-1}$, respectively. The method developed can be applied for the determination of inorganic elements in polypropylene, ice cream and acetic acid by ICP-OES.

Keywords Packaging · Ice cream · Inorganic contaminants · Migration · ICP OES

Introduction

Polymeric packaging is more commonly used to pack food and beverages. However, this type of packaging is not inert, and its composition may include potentially toxic chemicals that can migrate into food. Therefore, its use is controlled by regulators (Grob et al. 2008; Haldimann et al. 2013; Munoz et al. 2002; Reinas et al. 2012; Van Goetz et al. 2013; Vera et al. 2014).

The migration of a substance from a packaging into food depends on the chemical and physical properties of the substance, also on the food and on the packaging material. These may include the substance concentration, molecular weight, solubility, diffusion and partition coefficients between the packaging material and the food, the nature of the food and contact conditions between the packaging material and the food: time, temperature and contact area (Arvanitoyannis and Kotsanopoulos 2014; Kuorwel et al. 2013; Vera et al. 2014).

Inorganic additives, particularly the pigments and dyes added to polymers used to manufacture colour packaging, may contain lead (Pb), cadmium (Cd), mercury (Hg) and arsenic (As), which can migrate into food (Fordham et al. 1995; Zenebon et al. 2004; Cadore et al. 2008; Reimann et al. 2010; Özer et al. 2011).

In Brazil, pigmented polymeric packaging and the pigments used are regulated by Resolution RDC no. 52 (Brazil 2010), published by the Brazilian Health Surveillance Agency (ANVISA). In the European Union, no specific legislation exists for pigmented polymeric packaging. It must comply with the Regulation (EC) no. 1935/2004 (EC 2004) and Directive

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no. 10/2011 (EC 2011). Colourants used in plastics are covered by national measures. Certain colourants, in particular, cadmium pigments, are regulated by EU legislation on chemicals and listed in Annex XVII of Regulation (EC) no. 1907/2006 (EC 2006). They must comply with the general safety requirements of Article 3 of the Framework Regulation no. 1935/2004 (EC 2004) and are subject to risk assessment in line with Article 19 of the Plastics Regulation-Directive no. 10/2011 (EC 2011).

One of the foodstuffs sold in many different coloured packaging is ice cream, which is packaged mainly in polypropylene (PP) containers manufactured by injection molding, and then dyed in many different colours (white, orange, yellow, red, among others). In 2007, 113.4 million units of ice cream containers made of PP were sold in Brazil, which required the use of 5.225 metric tons of polymer (Datamark 2009).

For quantification of metals in polymers and food, the inductively coupled plasma optical emission spectroscopy (ICP-OES) technique provides high sensitivity and high accuracy, and the equipment is easy to operate. Its main advantage is that it provides simultaneous or sequential multi-element assessment. Consequently, assessment time is reduced, and the use of samples and reagents is minimized (Kiyataka et al. 2014; Butcher 2013; Ritter et al. 2004).

In the ICP-OES technique, the stage of sample introduction in plasma is very important because the size of the particle aerosol formed and selected by nebulizer and spray chamber influences the accuracy, precision and the limits of detection and quantification. The pneumatic nebulizers are more commonly used because they are easy to be employed, reasonably stable and have a low cost. The cross flow nebulizers used with spray chamber with double step (Scott) are fairly employed because they are designed to reduce the clogging problem, but they are less sensitive when compared with concentric pneumatic nebulizers with cyclonic chamber (Hou and Jones 2000).

Nowadays, sample preparation in closed vessels (e.g. microwave-assisted digestion and high-pressure digestion) is strongly favoured over open vessel or dry-ashing due to the many advantages gained (e.g. digestion times are shortened, a smaller amount of corrosive acid vapors is liberated and lower element loss by volatilization) (Krug 2008; Korn et al. 2008; Sneddon et al. 2006; Demirel et al. 2008; Suoranta et al. 2014). High-pressure digestion is an alternative system used to digest complex matrix. This technique has the advantage of reaching temperatures up to 320 °C in 40 min. The digestion internal pressure is 130 bar (1920 psi), allowing a greater mass sample to be digested, compared to the microwave technique; also, different samples may be digested at the same time (White et al. 1998).

Thus, this study set out to evaluate the concentration of Pb, Cd, Hg and As in ice cream packaging and to assess the migration of these elements into packaged ice cream using a high-pressure digestion system and ICP-OES. The specific migration of these elements into 3 % acetic acid was analyzed directly in the simulant by ICP-OES.

Materials and Methods

Samples

Ten samples of different colours of ice cream packaging made of polypropylene (PP) were collected in supermarkets in Campinas—São Paulo, Brazil, as described in Table 1. The ice cream flavour was not taken into consideration. Products with a production date no greater than 30 days were chosen, in order to minimize the influence of the time of contact in the specific migration assays.

Instrumentation

All the assessments of elements were performed using an ICP-OES, OPTIMA 2000DV model, from PerkinElmer Corporation (Shelton, CT, USA), coupled with a hydride generation and cold vapour system (Perkin Elmer-FIAS 400). The axial view was utilized, and the cold recombination area was removed with a shear gas interface. The whole system is controlled by the software *WinLab32™* (Perkin Elmer).

For the assessment of Cd and Pb, a Cross Flow pneumatic nebulizer and a Scott spray chamber were utilized. For assessment of As and Hg assessment, the hydride generation technique and the cold vapour generation technique were used, respectively, both using a solution of sodium borohydride (1.2 %) under alkaline conditions as reducing agent and simultaneous readings. The parameters used for this ICP operation are shown in Table 2.

For the decomposition of the samples, a high-pressure ash-er equipment, model HPA-S (Anton Paar GmbH, Graz, Austria) equipped with an aluminum rotor and a quartz digestion vessel with a 90-mL capacity was used.

Table 1 Descriptions of the analyzed PP packaging

| Samples | Food manufacturer ^a | Packaging colour | Capacity (L) |
|---------|--------------------------------|------------------|--------------|
| A | 1 | Blue | 2 |
| B | 1 | Green | 0.7 |
| C | 1 | Yellow | 0.7 |
| D | 1 | Red | 2 |
| E | 1 | Yellow | 2 |
| F | 2 | Gold | 0.9 |
| G | 3 | Pink | 2 |
| H | 3 | White | 2 |
| I | 4 | Yellow | 2 |
| J | 4 | White | 2 |

^a The same number correspond to the same food manufacturer

Table 2 Experimental conditions used on ICP-OES equipment

| | |
|---|---|
| Elements: Pb and Cd | |
| RF Power (W): 1300 | Main argon flow rate (L min ⁻¹): 15 |
| Torch configuration: axial | Auxiliary argon flow rate (L min ⁻¹): 0.2 |
| Sample flow (mL min ⁻¹): 1.5 | Nebulizing flow rate (L min ⁻¹): 0.55 |
| Integration and reading time (s): 10 | Wavelength (nm) Pb: I I 220.353 Cd: I 228.802 |
| Elements: As and Hg | |
| RF power (W): 1450 | Main argon flow rate (L min ⁻¹): 17 |
| Torch configuration: axial | Auxiliary argon flow rate (L min ⁻¹): 0.2 |
| Sample flow rate (mL min ⁻¹): 1.5 | Nebulizing flow rate (L min ⁻¹): 0.50 |
| Pump (rpm): 80 | Wavelength (nm) As: I 193.696 Hg: I 194.168 |

(I) Analyte line atomic; (II) analyte line ionic

Materials and Reagents

Deionized water (resistivity of 18.2 M Ω cm⁻¹) was obtained with a Milli-Q system (Millipore, Bedford, MA, USA). The glassware used in the present study was previously washed with extran detergent (Merck, Darmstadt, Germany), decontaminated by immersion in a 20 % (v/v) nitric acid solution for 12 h and then rinsed with deionized water. All chemical reagents were of analytical grade. Nitric, sulfuric and acetic acids, hydrogen peroxide and sodium hydroxide obtained from Merck and analytical grade sodium borohydride (Vetec, Rio de Janeiro, Brazil) were used. The standard stock solutions of Pb, Cd, Hg and As (1000 mg L⁻¹) were obtained from Merck. Stock solutions were diluted in HNO₃ (8 %), and 1.0 mL of H₂SO₄ (96 %) was added only in analytical curve for analysis in the polymer. Certified reference materials (CRM) on low-density polyethylene from the Institute for Reference Materials and Measurements (IRMM, Geel, Belgium), for ERM-EC681k and ERM-EC680k, were also used.

Sample Preparation

The digestion of the samples of PP packaging and ice cream was performed in a closed system, using HPA-S. Approximately 3 g of each sample (PP packaging) was cut into small pieces (1 cm \times 1 cm, approximately) and rinsed with deionized water. A 300-mg portion of each sample was then weighed and introduced into HPA-S equipment, closed, quartz decomposition vessel. To determine the digestion parameters, three digestion programs and three different mixtures of reagents were tested, resulting in five procedures (Table 3). The procedures were based in methodology described in Ritter et al. (2004); Sneddon et al. (2006);

Demirel et al. (2008); Korn et al. (2008); and Krug 2008. However, all cited authors used the digestion by microwave system, and this work used the high-pressure digestion system. For the initial tests, a white and a red sample of PP were used (D and H samples).

The ice cream sample was performed on day 0 (the date on which the sample was acquired) and after 365 days of storage at -20 °C. The ice cream being a solid food is expected that the migration of metals from packaging into food is in higher concentration on the surface of food. Thus, sample of surface ice cream was sampled and homogenized. After homogenization of ice cream, 1.0 g of each sample was weighed and introduced in a quartz decomposition vessel. In order to set the parameters for digestion, two different mixtures of reagents were tested (Table 3). After the digestion, all samples were diluted to a final volume of 25 mL with deionized water.

To determine the procedure for digestion of PP and of ice cream, the absence of opacity, supernatant and precipitate after the digestion were ascertained. In addition to the visual analysis of the digested material, the concentration of residual carbon was measured by ICP-OES, in order to ascertain the efficiency of the digestion of the samples, using the method described by Gouveia et al. (2001), and a standard curve was drawn based on a glucose solution on the 193.030-nm wavelength.

The specific migrations of Pb, Cd, Hg and As into 3 % acetic acid solutions as food simulant followed the methodology established by norm EN1186 (European Committee for Standardization 2002), according to Resolution RDC no. 52/2010 (Brazil 2010). The packaging was emptied and rinsed with deionized water and was kept in contact with the 3 % acetic acid solution at 40 °C for 10 days, using a standard ratio of area of material per volume of simulant of 6 dm² kg⁻¹, and after contact, an assessment of the elements was performed directly on the simulant by ICP-OES.

Validation Parameters

The validation parameters, linearity, limits of detection and quantification, accuracy and precision were carried out following the procedures described previously (Kiyataka et al. 2014) according to INMETRO (2011).

Results and Discussion

Evaluation of the Efficiency of Digestion

Method for Polymer (PP) Digestion

For the quantification of inorganic contaminants (Pb, Cd, Hg and the) in PP, the first step was the choice of the mineralization technique. The use of high-pressure furnace allowed the elimination of organic matter in a short period of time that was

Table 3 Procedures used in PP and ice cream digestion

| Procedures | Reagents | Step | Ramp time | Temperature | Duration |
|-----------------|---|------|-----------|-------------|----------|
| 1 ^a | 5 mL of HNO ₃ | 1 | 45 min | 250 °C | 30 min |
| | | 2 | 10 min | 320 °C | 120 min |
| 2 ^a | 5 mL of HNO ₃ | 1 | 45 min | 250 °C | 15 min |
| | | 2 | 15 min | 320 °C | 150 min |
| 3 ^a | 3 mL of HNO ₃ + 1.0 mL of H ₂ O ₂ | 1 | 45 min | 250 °C | 15 min |
| | | 2 | 15 min | 320 °C | 150 min |
| 4 ^a | 3 mL of HNO ₃ + 1.0 mL of H ₂ O ₂ | 1 | 45 min | 250 °C | 15 min |
| | | 2 | 15 min | 320 °C | 180 min |
| 5 ^a | 3 mL of HNO ₃ + 1,0 mL of H ₂ SO ₄ | 1 | 45 min | 250 °C | 15 min |
| | | 2 | 15 min | 320 °C | 180 min |
| 6 ^b | 2 mL of HNO ₃ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 10 min |
| 7 ^b | 3 mL of HNO ₃ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 10 min |
| 8 ^b | 2 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 10 min |
| 9 ^b | 3 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 10 min |
| 10 ^b | 3 mL of HNO ₃ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 30 min |
| 11 ^b | 3 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 30 min |
| 12 ^b | 3 mL of HNO ₃ + 1,0 mL of H ₂ O ₂ | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 30 min |
| 13 ^b | 3,5 mL of HNO ₃ | 1 | 5 min | 200 °C | 5 min |
| 14 ^b | 3,5 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 2 | 10 min | 320 °C | 30 min |
| | | 1 | 5 min | 200 °C | 5 min |
| | | 2 | 10 min | 320 °C | 30 min |

^a Procedures used in the digestion of PP packaging

^b Procedures used in ice cream digestion

efficient when combined with the use of appropriate reagents and digestion time, as shown in Table 1.

Procedures 1 to 4 were not efficient in completely eliminating organic matter. After digestion, the presence of a white precipitate and an opaque supernatant was observed.

Procedure 5 presented a colourless solution without supernatant and precipitate only for the red packaging. However, the concentration of residual carbon, measured after the digestion for both samples, was less than 1 %.

For the white PP packaging, despite the low level of residual carbon, the presence of the titanium dioxide pigment was noted in the samples. Thus, for the samples of white packaging, it was necessary to filter the digested sample with filter paper porosity of 2.5 µm in order to remove the titanium dioxide before the ICP-OES reading.

Method for Ice Cream Digestion

The residual carbon for procedures 10 to 14 were lower than 1.5 %, and the solutions after digestion were clear (Table 4).

Table 4 Digestion of the ice cream

| Reagents | Resultados | | |
|---|------------|--------|-------------------|
| | Procedure | Aspect | % Residual carbon |
| 2 mL of HNO ₃ | 6 | (1) | 7.7 |
| 3 mL of HNO ₃ | 7 | (1) | 4.1 |
| 2 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 8 | (1) | 6.3 |
| 3 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 9 | (1) | 3.1 |
| 3 mL of HNO ₃ | 10 | (2) | 1.2 |
| 3 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 11 | (2) | 1.1 |
| 3 mL of HNO ₃ + 1,0 H ₂ O ₂ | 12 | (2) | 1.2 |
| 3,5 mL of HNO ₃ | 13 | (2) | 0.9 |
| 3,5 mL of HNO ₃ + 0,5 mL of H ₂ O ₂ | 14 | (2) | 1.0 |

(1) Solution with supernatant

(2) Clear solution and not precipitate

Thus, procedure 13 was chosen, as the use of peroxide did not produce any significant improvement in the digestion of the ice cream samples.

Validation Parameter for Analysis of Inorganic Contaminants

Linearity

It was observed that the correlation coefficients are above 0.999 for all elements, suggesting a good linearity of analytical curves in the interval of concentration between 5.0 and 2000.0 $\mu\text{g L}^{-1}$ for the element Cd, between 10.0 and 2000.0 $\mu\text{g L}^{-1}$ for Pb and 1.0 $\mu\text{g L}^{-1}$ and between 15.0 $\mu\text{g L}^{-1}$ for both As and Hg.

Limits of Detection and Quantification

According to INMETRO (2011), for trace level analyses, it is recommended that the quantification limit be the lowest concentration on the analytical curve. For all the elements studied, solutions were prepared with the same concentration of the value calculated. For the elements Hg and As, concentrations lower than 1.0 $\mu\text{g L}^{-1}$ showed error greater than 20 %; thus, for the elements Hg and As, the lowest point on the analytical curve was adopted as the LOQ, 1.0 $\mu\text{g L}^{-1}$ (Table 5).

The limits of quantification (LOQ) expressed in micrograms (μg) per kilogram (kg) of polymer, considering the dilution factor of 83.3 times, were 1516.1, 216.6, 83.3, and 141.6 $\mu\text{g kg}^{-1}$ for Pb, Cd, Hg and As, respectively.

Recovery Study—Ice Cream

For the migration trials of Pb, Cd, Hg and As into ice cream, the recovery was assessed by adding three concentrations of each element to the strawberry-flavoured ice cream before

digestion. According to AOAC International (2013), the recovery assessment is a function of the concentration, and for a concentration of 10 $\mu\text{g kg}^{-1}$, there is an acceptable variation range between 70 and 125 %. Therefore, the method utilized was acceptable, given that the recovery of all the analytes in the ice cream was between 75.7 and 106.6 % (Table 6).

Recovery Study—3 % Acetic Acid

For the migration trials of Pb, Cd, Hg and As into 3 % acetic acid solution, the recovery was assessed by adding three concentrations of each element to the simulant. The specific migration method in 3 % acetic acid of the elements studied 7 was accurate. The variation in recovery of all the elements added to the 3 % acetic acid solution remained within the acceptable range of 70–125 % established by AOAC (2013) (Table 6).

Results for the Elements Pb, Cd, Hg and As in Polypropylene Ice Cream Packaging

Out of the ten samples analyzed, none of the packaging presented levels of As or Hg above the limit of quantification, and only the sample D the level of Pb was $1.64 \pm 0.64 \text{ mg kg}^{-1}$ and only the sample H the level of Cd was $0.45 \pm 0.03 \text{ mg kg}^{-1}$. The other samples showed levels of Pb and Cd below of the limit of quantification. Given that the positive list from Resolution RDC no. 17/2008 (Brazil 2008) contains no Cd or Pb-based additives, the only permitted source of these elements would be colourants used in the masterbatches for the manufacturing of the packaging. In Brazil, colourants and pigments are not included in the positive list from Resolution RDC no. 17/2008, but could be used, provided it meets the purity criteria established by Resolution No. 52/2010.

Table 5 Limits of detection and quantification

| Elements | Samples | Limits of detection (LOD) ($\mu\text{g L}^{-1}$) | Limits of quantification (LOQ) ($\mu\text{g L}^{-1}$) |
|----------|-----------------|---|--|
| Pb | PP container | 8.2 | 18.2 |
| | Ice Cream | 13.6 | 35.4 |
| | 3 % acetic acid | 6.7 | 26.2 |
| Cd | PP container | 1.4 | 2.6 |
| | Ice cream | 1.2 | 2.1 |
| | 3 % acetic acid | 1.3 | 4.9 |
| Hg | PP container | 0.3 | 1.0 ⁽¹⁾ |
| | Ice cream | 0.3 | 1.0 ⁽¹⁾ |
| | 3 % acetic acid | 0.5 | 2.1 |
| As | PP container | 0.6 | 1.7 |
| | Ice cream | 0.1 | 1.0 ⁽¹⁾ |
| | 3 % acetic acid | 2.2 | 8.4 |

⁽¹⁾The lowest concentration on the analytical curve was considered

⁽²⁾LOD = $X + t.s$;
LOQ = $(X + 10.s)$

X blank average, s standard deviation, t student

Table 6 Recovery of lead, cadmium, mercury and arsenic in the ice cream and in 3 % acetic acid solution⁽¹⁾

| Elements | Pb | Cd | Hg | As |
|--|-----------------|----------------|----------------|----------------|
| Ice cream | | | | |
| Added concentration ($\mu\text{g L}^{-1}$) | 50.0 | 25.0 | 2.5 | 7.5 |
| Recovery (%) ⁽²⁾ | 99.4 \pm 8.8 | 94.3 \pm 3.5 | 76.7 \pm 5.4 | 75.7 \pm 5.5 |
| 3 % Acetic Acid | | | | |
| Added Concentration ($\mu\text{g L}^{-1}$) | 50.0 | 10.0 | 2.0 | 6.0 |
| Recovery (%) ⁽²⁾ | 102.4 \pm 4.8 | 96.7 \pm 3.1 | 91.0 \pm 2.8 | 94.8 \pm 1.9 |

⁽¹⁾ Results of two assessments⁽²⁾ Average \pm standard deviation

Sample D, which presented level of Pb above the limit of quantification, is red; the most commonly used red dyes and pigments for polymers dying are lead, chromate, molybdate and sulfate (PbCrO_4 , PbMoO_4 and PbSO_4) (Cadore et al. 2008), which are substances classified as toxic to reproduction: category 1 by regulation EC 1907/2006 (EC 2006). In Brazil, lead-based pigments are accepted for contact with food as long as the quantity of migration into an extraction solution of HCl 0.1 mol L^{-1} is no greater than 100 mg kg^{-1} . That is why, it is necessary to demonstrate that the pigments utilized conform to the purity criteria established by Resolution RDC no. 52/10 (Brazil 2010). Because of the difficulty to get the pigments used in these packaging for inspection, the importance of establishing total limits for contaminants in the final packaging rather than in the raw material (pigment) is evident.

Sample H, which tested positive for Cd above the limit of quantification, is white; the white colourants most commonly used to give colour to polymers are titanium oxide (TiO_2), zinc sulfide (ZnS), zinc oxide (ZnO) and white lead ($\text{Pb}(\text{CO}_3)_2$). $\text{Pb}(\text{OH})_2$ (Cadore et al. 2008). These pigments do not contain Cd; so, the presence of Cd may be attributed either to a problem with the purity of the resin, of the pigment or of other additives used in this packaging. Annex XVII of Regulation (EC) no. 1907/2006 (EC 2006) establishes that cadmium-based pigments shall not be used to give colour to finished articles manufactured from the polymers (polyvinyl chloride, polyethylene, polypropylene and others).

Migration into Ice Cream

For all samples evaluated, the concentrations of the elements Pb, Cd, Hg and As on day 0 were below the limit of quantification (LOQ). After 365 days of storage at -20°C , no migration from the packaging to the food above the LOQ of the elements studied was detected. Therefore, the Pb and Cd present in samples D and H did not migrate to the ice cream.

Results of the Specific Migration Test in 3 % Acetic Acid

The results obtained in the study of the migration of the elements Pb, Cd, Hg and As into 3 % acetic acid solution were below the limit of quantification.

The levels of the elements Cd, Hg and As that migrated from the PP packaging into an aqueous solution of 3 % acetic acid were below the limits established by Resolution RDC no. 52/10 (Brazil 2010), which are 5, 10 and 5 $\mu\text{g L}^{-1}$, respectively.

However, for LOQ of Pb was above the limit established by Resolution RDC no. 52/10 (Brazil 2010). Kiyataka et al. (2014) used a Miramist pneumatic concentric nebulizer (Burgener Research Inc., Mississauga, ON, Canada) with a quartz cyclonic spray chamber in the determination of specific migration of Pb into a solution of 3 % acetic acid and found a

limit of quantification lower than $10 \mu\text{g kg}^{-1}$. Therefore, sample D was retested using a nebulizer Miramist with a quartz cyclonic spray chamber, and the result for specific migration of Pb into a solution of 3 % acetic acid was below the limit set by Resolution RDC no 52/10. Thus, applying a nebulizer with cyclonic chamber quartz by ICP-OES is recommended in the determination of specific migration of Pb into solution of 3 % acetic acid.

Conclusion

The proposed methods of decomposition of the PP packaging and ice cream showed to be efficient. The procedures presented adequate sensitivity, precision and accuracy. Addition and recovery studies indicated that the method developed can be applied for the determination of inorganic elements in PP polymers, ice cream and 3 % acetic acid by ICP-OES. The specific migration of lead into a solution of 3 % acetic acid using a Miramist nebulizer is needed for the LOQ to be appropriate. None of the sample packaging analyzed presented levels of As or Hg above the limit of quantification, and only one sample showed results of Pb above the limit of quantification. Another sample presented concentration of Cd above the limit of quantification, which points to the use of pigments and additives containing these elements. Given the difficulty to inspect the pigment utilized in these packaging, it is important to set limits of inorganic contaminants in the final packaging. The results of migration of Pb, Cd, Hg and As into 3 % acetic acid were below the limit set by Brazilian Legislation. There was no migration of Pb, Cd, Hg and As into ice cream above the limit of quantification. Although ANVISA recommends using a simulant for the study of migration, it is worth underscoring that the migration testing in food will consider specific and intrinsic parameters that are important to the study of migration.

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Conflict of Interest Paulo Henrique Massaharu Kiyataka declares the he has no conflict of interest. Sílvia Tondella Dantas declares the she has no conflict of interest. Juliana Azevedo Lima Pallone declares the she has no conflict of interest. This article does not contain any studies with human or animal subjects.

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